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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at 61 New Cavendish Street, London, W.1, on 3 December 1958, the Chair being taken by C. M. Vignoles, C.B.E., President of the Institute.

The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting.

The Chairman introduced the authors and the following papers were presented in summary.

FLOW PROPERTIES OF DISTILLATES AT LOW TEMPERATURES: A REVIEW *

By J. F. HUTTON †

SUMMARY

A critical review is given of the conventional tests for assessing the lower temperature limits of use of distillate fuels and lubricants, namely, cloud point and pour point. Evidence is presented to show that these tests should not be used as criteria of flow properties. This conclusion applies particularly to laboratory investigations aimed at finding rules for the proper selection of additives for increasing the fluidity of oils at low temperatures.

Mechanisms by which the additives increase fluidity are also reviewed, bearing in mind that much of the published information has been directed towards the study of cloud and pour point depression. It is concluded that the most likely mechanisms are: (1) surface adsorption, and (2) change of crystal habit and size by co-crystallization. By these mechanisms inter-particle attraction is reduced. The third possibility, namely, the formation of eutectic mixtures, is not considered to be important. This mechanism would result in a lowering of the crystallization temperature.

INTRODUCTION

PROBLEMS that are attributable to a sudden increase in resistance to flow arise during the use of fuels and lubricants at low temperatures. Thus, oils solidify in pipelines or filters block. The problems in mind are known, or suspected, to be caused by the precipitation of wax crystals from the oil. In wet oils at low temperatures ice crystals also hinder flow, but this aspect is not considered here.

It is not the aim of this paper to review the literature on the low temperature flow properties of all types of fuels and lubricants. The discussion is limited to distillates. This means that oils containing appreciable quantities of asphaltic material are excluded. A characteristic of oils containing asphaltenes is that their behaviour at low temperatures depends considerably on their thermal history. Distillates show this effect only slightly or not at all. The complications of thermal history effects in oils containing asphaltenes have led to the initiation of considerable research into test methods, mechanism studies, and oil treatments for these products. On the other hand, with distillates the problems have appeared much simpler. Test methods have been devised and oil treatments developed which have adequately met all demands. Consequently, basic studies of the low temperature flow properties of distillates have been minimal, and very little has been published.

Nevertheless, there is an increasing awareness that the research effort on distillates should be expanded. Let us consider, for example, diesel fuels. The ignition quality of a diesel fuel is improved by increasing the paraffins content, but paraffins cause the fuel to gel at relatively high temperatures. Therefore a compromise must be found, which means in practice a search for preferred blending components. Perhaps special blends will have to be made for particularly difficult markets. A result of this course of action is a lack of flexibility in the blending programme. In the future, when more and more new products are taken from a gallon of crude oil and greater demands are imposed on products, inflexibility in blending programmes will be tolerated less and less.

In our example, fluidity at low temperatures could be increased by the use of an additive, but economic considerations would exclude all but cheap additives, or additives which are effective in extremely small proportions. Also, the additive, or mixture of additives, should be effective in as many as possible of the components used to blend a range of fuels, *i.e.* it should not be too specific in its action. Therefore, the problem is reduced to one of additive selection. Nearly all the work done on the type of additive required has been based on the effect additives have

* MS received 18 November 1958.

† "Shell" Research Ltd.

on the cloud point and pour point (ASTM method D97-47; IP method 15/55). Few, if any, additives affect cloud point, but there are a vast number which are known to depress the pour point. However, very few rules exist for the proper selection of the so-called pour point depressants, so the solution to our example problem, in the present state of knowledge, must be obtained by a trial and error approach.

In this paper the two standard tests are critically discussed, and the suggestion is advanced that a reason why there are so few rules which can be applied in the choice of effective additives for improving fluidity at low temperatures lies in the use of these standard tests as criteria. The extent of our knowledge of the action of additives is outlined in the light of this suggestion.

THE STANDARD TESTS FOR CLOUD POINT AND POUR POINT

During continuous cooling an oil first develops a turbidity while still remaining fluid and secondly gels to a rigid mass. These phenomena are caused by wax crystals which start to come out of the oil solution at a certain temperature. As the temperature is lowered, the concentration of solid material gradually increases until there is sufficient to form a wax network structure.

Cloud Point

The usual test for initial wax separation is the cloud point. In this test, carried out in standardized conditions, the temperature at which a visible haze appears is noted. Since a visible haze requires a fairly large quantity of solid material, the cloud point will always be lower than the true limiting temperature for complete solution of the wax present. Now a given oil will, in general, contain a variety of waxes of different chain lengths and molecular types present in various concentrations. Thus it is possible that an oil will contain traces of high melting point (and therefore low solubility) wax which will not be detected visually in the cloud point test. Therefore, not only will the " true " cloud point be higher than the observed value but it can also be much higher. Also, the difference between " true " and observed values will in general not be the same for different oil samples.

It is usually considered that the cloud point is related to the blocking of filters by fuels. Thus, at temperatures below the cloud point a filter will block. This may be true to some extent—although exceptions might be expected, depending on the strengths of adhesion between wax crystals and the walls of the filter holes, and between the first layer of crystals and the next; but it follows from the above argument that the converse cannot be true: that at temperatures above the cloud point a filter will not block. In fact, it has been observed that fuel filters can be blocked with waxy material at temperatures well above the measured cloud point, sometimes only after many hundreds of gallons of fuel have been passed through. This result suggests the presence in the fuel of trace quantities of high melting point wax.

Alternative tests to the cloud point must detect very small concentrations of wax. The use of a microscope will give a closer value to a " true " cloud point, but this method too will have a concentration limit. The most reliable method, which would be too slow for other than research and development purposes, is a large-scale filtration test carried out at various temperatures down to below the cloud point.

A compromise between a large-scale filtration test and a rapid laboratory test has been developed in Germany. In the DIN 51770 filterability test the time is measured for a volume of 200 ml of oil to pass through a standard filter at constant temperature. The test is repeated at successively lower temperatures until the measured time interval exceeds 60 seconds. The temperatures corresponding to the 60 seconds time interval is the filterability limit. In addition to this temperature the test gives a curve of times against temperature which can also be used to compare the performance of oils.

Pour Point

The usual test for gelation of an oil is the pour point. The determination is carried out by cooling the unstirred oil in stages by successive immersion of a jar of oil in progressively colder constant temperature baths. At intervals of 5° F (2·8° C) the jar is removed from the bath and tilted to detect flow. Eventually a temperature is reached when, with the jar tilted horizontally, so that the oil surface is vertical, the oil is observed to show no movement in 5 seconds. The pour point is 5° above this temperature and is said to be reproducible to $\pm 5°$.

Rheologically this test is not very sound, because although the shear stress distribution at the pour point is much the same in all tests (the stress is a function of the height of oil and, therefore, of the jar diameter), the rate of shear is bound to be variable because it is assessed subjectively. To illustrate this point, we consider the published reports concerning the so-called viscosity pour point. This is measurable in wax-free oils and is a temperature at which the oil viscosity has reached a high value. Gruse and Stevens [1] say the viscosity at this temperature is about 5×10^4 stokes or about 5×10^4 poise. Gavlin *et al* [2, 3] report values between 1×10^3 and $1{\cdot}4 \times 10^3$ poise. Thus, there is a difference up to 50-fold in the estimation of shear rate in the experiments.

Another illustration is provided by reports that changes in size of the test jar or of the volume of the sample do not affect the measured pour point. For

instance it has been found [2] that pour points measured on samples contained in very small vials of about 2 ml capacity are identical with the normal values measured on the standard volume of about 40 ml. The result is also quoted and shows that the pour point is independent of sample size for quantities of oil 125 ml or less. These observations imply that the pour point is independent of shear stress over a wide range of shear stress. This will be effectively true if the oil develops a rigid structure within a narrow temperature range. However, Bondi [4] has shown that whilst a straight oil may show a marked rise in apparent viscosity (indicative of a sharply defined yield stress) just below the pour point, the same oil with an additive shows only a gradual rise in viscosity through the pour point. If, as appears to be the case, oils such as the one containing additive have a pour point independent of sample size, it indicates that the rheological conditions of the test are badly defined. As a consequence, ambiguities in assessing flow properties at low temperatures can arise.

Daniel's data [5] appear to agree with Bondi's on this point, as do those of Davis and Zimmer.[6] Davis and Zimmer state that the pumpability limit of (straight) oils is 10° to 15° F below the pour point, but with Paraflow present the pump still operates at lower temperatures than 15° below the pour point. Strawson [7] also finds a measure of correlation between pumpability limit and pour point for certain kerosines, whilst for others (which may contain natural depressants) the correlation fails.

The following illustrates the insensitivity of the pour point test. In a Russian method [8] a metal plate is withdrawn from a gelled oil at a slow constant rate. The stress required first rises linearly and then passes through a maximum (P). The value P can be considered as a yield limit. It was found,[9] using C30 n-paraffin in a wax-free oil, that additions of sodium dioctyl sulphosuccinate and diethylene glycol dilaurate lowered P considerably. Paraflow and Santopour lowered P even more. However, in some unpublished experiments with similar " ideal " waxy oils, only the last two additives were found to be effective pour point depressants. The two synthetic detergents did not significantly change the pour point. Consequently, in studies of the mechanism of pour point depression the synthetic detergents would be classified with ineffective additives. Because of this lack of sensitivity, valuable correlations between other measurable properties of additives and their effectiveness in increasing fluidity may be lost.

It is concluded that the pour point test should not be used in research and development work as a criterion of low temperature flow behaviour. At best it will give a rough measure of changes in fluidity if the differences between oils are very large. In fact, differences in pour point of less than 10° F are within the reproducibility of the test and are not significant; yet in an oil a real improvement of such a magnitude is of considerable benefit. Alternative tests to the pour point must be fully quantitative and should be based on sound rheological principles. These conditions can be met by making measurements of the shear stress/rate of shear curve as a function of temperature and, in certain cases, as a function of total amount of shear. Such measurements will obviously take much longer than a pour point test, but, as more is learned of the rheology of wax gels, new simple forms of test may emerge.

As a direct replacement of pour point a yield stress measurement should be made. Necessary conditions for yield stress determinations are the application of very low rates of shear and very small total shears. Depending on the design of instrument used, it may also be necessary to avoid slip effects at the instrument wall. An example of a type of yield stress measurement is used in the work described by Strawson [10] in the accompanying paper.

The viscosity, *i.e.* the ratio of shear stress to rate of shear, should be measured at temperatures both above and below the solidification point. Above solidification the viscosity will be practically independent of rate of shear, and simple instruments can be used. Bondi has shown [4] that additives change the slope of the viscosity/temperature curve in this region. Comparisons of these viscosity/temperature curves will be more informative and less ambiguous than comparisons of pour points. In the vicinity of and below the solidification temperature, viscosity will depend on rate of shear and amount of shear, as has been shown by experiments carried out in the U.S.A.[3] The rate at which viscosity falls with amount of shear (*i.e.* with time at constant rate of shear) will be a measure of the permanence of the gel structure, whilst the equilibrium value will be the viscosity of the oil when the structure is completely broken down. The U.S. work has shown that the equilibrium viscosity is a little higher than that to be expected for the wax-free oil at the same temperature, and probably obeys the Einstein equation for a non-interacting suspension

$$\eta = \eta_0(1 + 2{\cdot}5C) \quad . \quad . \quad . \quad (1)$$

where η is the equilibrium viscosity, η_0 is the viscosity of the wax-free oil, and C is the volume concentration of the discrete wax particles. Just below the pour point C is only about 0·01 to 0·03.

In an application where the oil is always agitated either by motion of the reservoir or by the presence of a totally immersed pump, the controlling factor in pumpability will be a viscosity close to the equilibrium value. Yield stress and pour point will have only a minor significance. Hammerich and Schmitz [11] consider that the starting limit for a motor oil is the temperature at which the viscosity reaches 1000° Engler or about 8000 cS. It appears from the form of their results that the viscosity referred to is the

equilibrium value. Their results show that the starting limit is quite independent of pour point (strictly the *Stockpunkt*, DIN 51583). With some of their oils the starting limit is above the pour point, whilst with others it is below the pour point. A similar conclusion has been drawn from some Polish work.[12] In this study the limiting value of viscosity (for satisfactory flow through the passages in an engine) was taken to be 8000° Engler.

So far we have dealt with the shortcomings of the standard cloud point and pour point tests and have outlined the principles on which new test methods should be based. Now considerable effort has been devoted to studies of the mechanism of pour point depression. In these studies the assumption is made that the pour point is a well-defined characteristic of an oil and a characteristic which governs the low temperature flow properties. In view of the criticism we have made of pour point, it is pertinent to consider whether the current views of the mechanism of pour point depression can be adapted to the more general concept of the mechanism of increasing fluidity at low temperatures. This task is attempted in the following section.

MECHANISMS OF THE FORMATION AND SUPPRESSION OF WAX GEL STRUCTURES

Formation

The wax precipitation from oil fractions is in general a mixture of *n*-paraffins, *iso*-paraffins, and cyclic hydrocarbons.[1] More specific information is given in a Russian paper.[13] Here it is shown that the crystallization of a fuel is determined mainly by *n*-paraffins but also by bicyclic aromatic hydrocarbons having not more than two carbon atoms in their side chains. For fuel fractions consisting of monocyclic aromatic or monocyclic naphthenic hydrocarbons boiling between 150° and 350° C, or bicyclic aromatics of longer side chain length than two carbon atoms, crystallization does not occur. At low temperatures these fractions transform into an amorphous, glassy state.

At the pour point of an oil the amount of wax precipitated is about 1 to 3 per cent of the solution. This is only a small quantity compared with, say, the amount of soap required to gel an oil to form a grease. Therefore, the attractive forces between the wax particles must be high. The high forces owe much to the small particle size of the wax. The probability of particle collisions, and hence of particle adhesion, for a given volume of solid is inversely proportional to the average radius to a high power.[4] Therefore, a rapidly-chilled oil, giving small crystals, will gel at a higher temperature than a slowly cooled oil. The fact that the wax crystals have a plate-like habit [14] also assists gelling by reducing the distance over which the forces have to act. The alignment of particles in the structure is then edge-to-face like a house of cards. Immobilization of the oil phase by solvation of an oil layer around each particle can also contribute to gelling.

Suppression

Very little work has been done on the improvement of low temperature flow properties without the use of pour point as the test criterion. Therefore this section will deal largely with general theory and results which have not involved pour point measurements.

Suppression of the wax structure might be achieved in a number of possible ways and combinations of these ways:

(*a*) increasing wax solubility;
(*b*) change of crystal habit to a more isometric shape;
(*c*) method (*b*), together with an increase in crystal size;
(*d*) reduction of attractive forces by an adsorbed layer of surface active material.

The basis of mechanism (*a*) is that the wax and additive and the wax/additive/oil systems form eutectic mixtures which have a lower crystallization temperature than the pure wax. Experimental evidence is in general against the eutectic theory, although work carried out in Poland [12] has led to the conclusion that Paraflow acts by forming eutectic mixtures, whilst Santopour acts mainly by a surface mechanism.

Evidence against the eutectic theory is provided by X-ray diffraction and thermal analysis data. Thus, Gavlin, Swire, and Jones [3] show that the X-ray diffraction pattern of the wax precipitated from an oil is unchanged if a pour point depressant is added to the oil. They conclude that the additive must be on the surface of the wax crystals. The thermal data obtained by Bondi [4] show that for waxy oils the crystallization temperature range and the associated latent heat are unchanged by the addition of pour point depressants. The ideal solubility equation is

$$\ln S = (L/R)(1/T - 1/T_0) \quad . \quad . \quad (2)$$

where S is the fractional solubility at absolute temperature T, L is the latent heat of fusion of the wax, T_0 is the melting point of the wax, and R is the gas constant. Therefore if, as Bondi found, T_0 and L remain unchanged by additives, the solubility relationship is unchanged. Thermal analysis experiments by Gol'denberg and Zhuze,[15] carried out on pure *n*-paraffins with and without additive, show a fall in crystallization temperature of only 1° or 2° C when the concentration of Santopour is increased from zero

to 30 per cent. Therefore, in solutions of the *n*-paraffins, the contribution by a eutectic mechanism to the large effect of Santopour on fluidity must be correspondingly very small.

Regarding mechanisms (*b*) and (*c*) (change of crystal habit and size) the evidence is confusing. Some workers,[2, 6] on the basis of microscopic evidence, report that with an additive present the wax crystals are smaller and more compact (isometric) than without additive. Daniel [5] has found that, provided the rate of cooling is the same in the comparative experiments, the additive has no effect on crystal size. Unpublished work known to the author has shown that in some systems pour point depression is associated with a change to larger, more isometric particles. These divergent results might be due to different mechanisms operating in the different systems studied or to misinterpretation of the microscope observations. Rhodes, Mason, and Sutton,[14] and Edwards [16] have studied the growth of wax crystals. They have found that under all conditions wax crystals grow initially in the form of plates, but in certain circumstances the plates roll up partially or completely to form needles, whilst in others the plate form is stable. It is thus easy to see how misinterpretation of size and shape changes can arise.

If a change of crystal shape to a more isometric form does occur it can be interpreted on the theory that co-crystallized additive molecules provide nuclei for the formation of additional layers. Wax crystals grow in a plate-like form because the rate of growth in directions normal to the large faces is slow relative to the rate normal to the edges. The modern conception is that the large faces are nearly perfectly formed crystal planes with very few nuclei which can initiate the growth of the next plane. Therefore, if an additive molecule has a side chain which co-crystallizes with the wax the rest of the molecule provides an additional nucleus for the next layer. It might be deduced from this theory that the co-crystallization phenomenon would require an additive of similar solubility characteristics to that of the wax. If this deduction is correct it would be very difficult to explain why some polymers that are soluble in oil down to very low temperatures, compared with the wax precipitation temperature, are effective in modifying the wax gel structure. However, the solubility requirement may not be valid. In the crystallization of ionic crystals from aqueous solution habit modification by added soluble dyes is in no way related to the solubility of the dyes.[17, 18]

Borrowing again from experience in crystallization from aqueous solution, it appears unnecessary that the co-crystallizing additive should have a structure similar to that of the salt.[17, 18] Therefore, the fact that a considerable number and variety of materials have been proposed as pour point depressants does not conflict with the co-crystallization theory.

Yet again, the X-ray diffraction pattern of a modified crystal can remain identical with that of the unmodified one.[19] A similar result for wax crystals has already been mentioned. Therefore, this result also does not conflict with the co-crystallization theory.

Lastly, we consider mechanism (*d*), which is linked with the co-crystallization mechanism in that adsorption must occur as the first step in either process. However, in mechanism (*d*) further crystallization will not occur on the adsorbed layer. The layer will reduce inter-particle attractive forces by a shielding action.

Gavlin, Jones, and Swire [20] claim that their results do not support this mechanism, since the irreversible mechanical breakdown of a wax gel to a suspension of discrete wax particles would not be expected if the gel structure had been held together by electrical forces. But in other work [3] the same authors show that not all wax gel structures break down irreversibly. The wax gel structures studied by Daniel [5] also exhibit reversibility. A possible explanation of the irreversible effect is that surface active material naturally present in the oils concerned adsorbs on the wax particles over the areas where a junction has been ruptured in shear, and thus prevents reformation of junctions when the shearing forces have been removed. (In the original formation of the gel the relatively slow rate of diffusion of the surface active material, compared with the rate of wax crystallization, would permit inter-particle adhesion to occur.)

Daniel [5] prefers the adsorption mechanism because of his negative results on crystal modification. Experiments carried out by Bondi [4] show that Paraflow fails to depress pour point when the oil is chilled rapidly. Bondi calculates that for the amount of Paraflow present in his oil samples there is insufficient active ingredient to fully coat all the surface area of the small wax particles produced by chilling. Therefore he concludes that the additive acts by surface adsorption. However, the results conform also with a co-crystallization mechanism if it is assumed that the rate of diffusion of the large Paraflow molecules in the oil is less than that of the wax molecules. Davis and Zimmer [6] consider adsorption to be more important than the crystal modification which they find accompanies pour point depression, since crystal modification has also been observed in the absence of pour point depression. Ruhrwein [21] agrees with their experimental evidence.

Very little fundamental work has been done in studying the adsorption mechanism because of the experimental difficulties. The difficulties arise because of the small changes in work of adhesion to be expected when surface active materials are added to an oil. In a suspension of wax particles in oil there is a competition between the tendency for particles to adhere together and the tendency for them to be completely surrounded, or wetted, by oil and thus

remain as discrete particles. The work of adhesion W between oil and wax particles is given by [22, 23]

$$W = \gamma_o + \gamma_w - \gamma_{ow} \quad . \quad . \quad . \quad (3)$$

where γ_o and γ_w are the surface tensions (surface free energies) of oil and wax respectively and γ_{ow} is the interfacial tension between oil and wax.

When additives are dissolved in an oil the surface tension will remain virtually unchanged,[23] but γ_{ow} will decrease by an amount depending on the surface activity of the additive. Thus the greater the surface activity the greater the reduction in γ_{ow}. Then the smaller is γ_{ow} the greater will be W, and the greater will be the tendency for oil to wet the wax particles and thus keep them separate.

Now because oil and wax are of similar nature it would be expected that γ_{ow} will be small, so any changes in γ_{ow} will also be small. Therefore, direct measurement of this surface parameter and changes in it will be difficult.

Apart from these surface considerations it may be expected that separation of wax particles will be influenced by the geometry of the adsorbed layer. Thus, if the layer is thick and bulky the particles may not approach close enough to interact and adhere. A suggestion that this mechanism is operating is found in some Russian work.[15] Thus, it is found that surface active materials of low molecular weight, or even of high molecular weight, provided the molecule has a straight chain structure, do not affect the crystal habit of *n*-paraffins. (Change in habit from plates to needles is found to accompany increased fluidity of gels.) On the other hand, materials of higher molecular weight (800–1000), and having a complex structure, cause radical changes in crystal habit. However, in the absence of direct measurements of surface properties this result cannot be considered as conclusive evidence of geometrical hindrance caused by bulky adsorbed molecules.

A few experimental studies have been made of the adsorption mechanism. Thus, Davis and Zimmer [6] show that the amount of Paraflow concentrated in a wax precipitate follows a typical adsorption isotherm. Fox and Zisman [24] show that the polycrystalline paraffin wax surface may have different wetting properties in different parts, depending on whether the molecular groups in the surface are methyl or methylene.

Conclusions

By the evidence and arguments presented in this section, we are led to the conclusion that none of the postulated mechanism is definitely eliminated. However, the lowering of the crystallization temperature by the formation of eutectic mixtures has at most a minor effect in the systems studied. The reduction of inter-particle attractive forces by crystal modification or by surface adsorption of additive molecules are the most probable mechanisms by which fluidity at low temperatures may be increased.

DISCUSSION

During most of the long history of the cloud point and pour point tests the degree of correlation between the measurements on distillate oils and the performance of the oils at low temperatures has been sufficiently high to permit satisfactory selection and control of blends. However, with the present increasing complexity of refinery programmes, more meaningful tests will be necessary. For the filter blocking problem a preferred test would detect much smaller quantities of solids than does the cloud point test. For problems associated with wax structure formation, yield stress and viscosity measurements should replace the subjective single-value measurement of pour point.

The facts that the pour point test is rheologically unsound, is insensitive, and provides very limited information may account, in part, for the existence of so few rules for the proper application of additives. However, we should be exceedingly rash to think that improved tests would lead directly to a complete set of rules. It has been pointed out above that even in relatively simple crystallizing systems crystal modifiers are very specific in their action. Nevertheless, it does appear that studies of pour point depression, which have been continuing for many years, have reached the limit of their usefulness. It is suggested that advances in the development and study of additives will be effectively made only by the development and use of laboratory and rig tests having a closer relationship with actual flow problems.

Finally, it should be pointed out that as long ago as 1943 Bondi [4] argued that the pour point test was inadequate for defining low temperature properties, but apparently it was then not considered an urgent matter to devise new tests. For since that time few published papers have given the results of rheological measurements on oils gelled by wax. Perhaps the present time is more opportune to devote more effort to these problems.

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THE PUMPABILITY OF AVIATION TURBINE FUELS AT LOW TEMPERATURES *

By H. STRAWSON †

SUMMARY

A study of the factors which limit the use of fuels at very low temperatures in turbine-powered aircraft shows that a fuel can be handled satisfactorily up to the point when partial solidification prevents adequate flow within the fuel tank itself. The lowest fuel temperatures which will be met in practice are assessed. It is shown that the fuel properties laid down in existing turbine fuel specifications to ensure adequate low temperature performance are not directly related to the problem, and that there is a need for an improved specification test for pumpability. Such a test method is proposed.

Finally the possibility of improving the low temperature performance of fuels by the use of additives is discussed.

INTRODUCTION

With the great expansion which is taking place in the use of turbine-powered civil aircraft, particularly those flying for long durations at high altitudes, the provision of kerosine type fuels (*i.e.* those having flash points above approximately 100° F), which can be used at the low temperatures to be encountered has become a question of considerable importance to airline operators, aircraft manufacturers, and fuel suppliers.

As the temperature is lowered the significant fuel properties which alter are the water solubility, the viscosity, and the extent to which solid hydrocarbon material precipitates (*i.e.* freezing of the fuel). It is the limitations imposed by this last process which form the subject of this paper, but the way in which the limitations apply is, in fact, modified indirectly by the first property, water solubility.

The reason for this is as follows. At temperatures below 0° C most of the water which has been dissolved in the fuel at higher temperatures is thrown out of solution, usually as supercooled water droplets, and can lead to the blockage by ice of the fine filters used to protect the engine fuel system. Although this problem is often alleviated by natural drying of the fuel as the aircraft climbs, such a phenomenon cannot be relied upon, so that it is now a universal practice in civil airliners to provide means for heating the fuel prior to the filter. It can therefore now be assumed that the fuel downstream of this point will not be subjected to particularly low temperatures in flight. This is important, because the filters used are very rapidly blocked by solid hydrocarbons (or wax) precipitated from fuels, and in the absence of fuel heating, filter waxing would impose a definite limit on low temperature handling.

With the use of fuel heating, however, the low temperature problem in the aircraft resolves itself into that of extracting fuel from the aircraft tanks and delivering it to the engine. In this light the paper discusses the flow processes involved, and the rheological properties of the fuel which are significant. An estimate is then made of the temperatures which will be experienced during flight, to indicate to what extent existing fuels will be suitable. Possible methods of improving the situation are suggested.

Only the requirements of medium- and long-range civil turbine-powered aircraft are considered. On the military side maximum availability is required, so

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† "Shell" Research Ltd.

that the principal demand is for a wide range fuel, where pumpability at very low temperatures presents no problem. Where other fuels are called for, the requirements are too specialized to be included in this paper.

LOW TEMPERATURE PUMPABILITY

The Aircraft Fuel System

Accepting that the problem is one of extracting fuel from the aircraft tanks and delivering it to the engines, these processes will be dealt with in some detail.

The tanks may be rubber bags carried in the wings, or may be formed integrally with the wings, sections of which are made fuel-tight during construction. In either case it is a universal practice to provide an electrically-driven impellor type pump, known as a

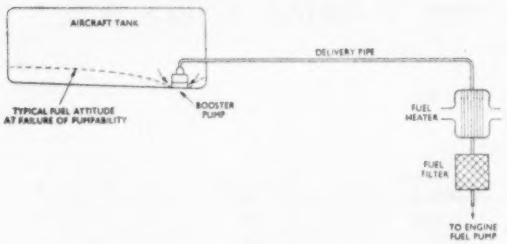

FIG 1

DIAGRAM OF AIRCRAFT TANK AND FUEL DELIVERY SYSTEM

" booster pump," at or near the lowest point of each tank, or each group of interconnected tanks. The booster pump delivers fuel to the engines and develops sufficient pressure (of the order of 15 p.s.i.) to overcome pipe friction and the pressure drops across the fuel heater and fuel filter, whilst maintaining adequate pressure at the inlet to the main engine fuel pumps and control system. Fig 1 shows the essentials of a typical system in diagrammatic form.

In the process of pumping fuel from the tank to the fuel heater, therefore, four main flow regions can be distinguished: (1) flow upstream of the booster pump, *i.e.* from the various parts of the tank to the pump inlet; (2) flow into the pump, through a coarse mesh screen; (3) flow through the pump itself; and (4) flow through the pipelines downstream of the pump.

The different flow conditions in these cases are influenced by different aspects of the fuel's rheological properties. Due to the large cross-sectional areas involved, the flow upstream of the pump normally involves very low rates of shear, the highest occurring when the tank is nearly empty and the fuel has to flow along the tank bottom, or in certain cases where interconnecting pipes are used between individual tanks of a group, or where the pump itself is separated from the tank by a relatively narrow channel. The shear forces available to cause flow are likewise very small, being due entirely to gravity.

At the pump inlet the head of the fuel will be supplemented by the prevailing atmospheric pressure. For instance, at 45,000 ft altitude the pressure is 2·1 p.s.i., and since the vapour pressure will be negligible at the temperatures in question, this pressure will be available to force fuel through the gauze screen at the pump inlet. Within the pump the fuel is, of course, subjected to very high rates of shear.

Downstream of the pump shear rates are moderate, as the fuel flows through the delivery pipes to the engines. There are, however, usually sections of pipe in which the fuel may be stagnant for part of the flight, in particular cross-feed pipes interconnecting the fuel systems of both wings, or individual engines.

Low Temperature Performance of Fuels

To examine the rheological properties of fuels which control their pumpability after solidification has commenced, two types of laboratory test have been used. First, the general relationships between shear stress and rate of shear have been examined at various temperatures in a conicylindrical viscometer. This instrument, which was designed to apply a constant shear stress irrespective of shear rate, demonstrated the highly thixotropic nature of the fuels under these conditions. For example, Fig 2 shows a plot of shear rate against shear stress for reference kerosine SR 312 at three temperatures, −56·3° C, −58° C, and −60·3° C. The freezing point and pour point of this fuel were −43° C and −57° C respectively. At −56·3° C the fuel is only slightly thixotropic and possesses a small yield value of approximately 5 dynes/cm². At −58° C the yield value is higher and the thixotropy marked. The line for falling stress, *i.e.* after breakdown of the structure, approximates closely to that at the higher temperature. At −60·3° C the thixotropy is even more marked and the falling line is still almost coincident with those at the higher temperatures, showing that the structure has been almost completely broken down.

A property demonstrated by these curves is the very distinct yield value above which small increases in shear stress give large increases in shear rate; in other words, providing that the force available is only moderately greater than that required to initiate flow, the rate of flow is likely to be adequate. For this reason, in estimating whether or not a fuel will flow, either in the tank or along a pipe, the most important property to determine is its yield value. This is simpler than obtaining the full shear stress/rate of shear relationship over a range of temperatures, and can theoretically be accomplished by measuring the pressure required to force fuel through a length of small-bore tube within which it has been cooled to the desired temperature. Yield value tests of this type have been used in comparing different fuels but, as mentioned later, difficulty has been experienced in achieving accurate results.

The performance of fuels in the four regions of flow mentioned in the previous section has been investigated at Thornton Research Centre by observing their behaviour in actual aircraft fuel systems installed in cold rooms, as well as by laboratory tests of their rheological properties.

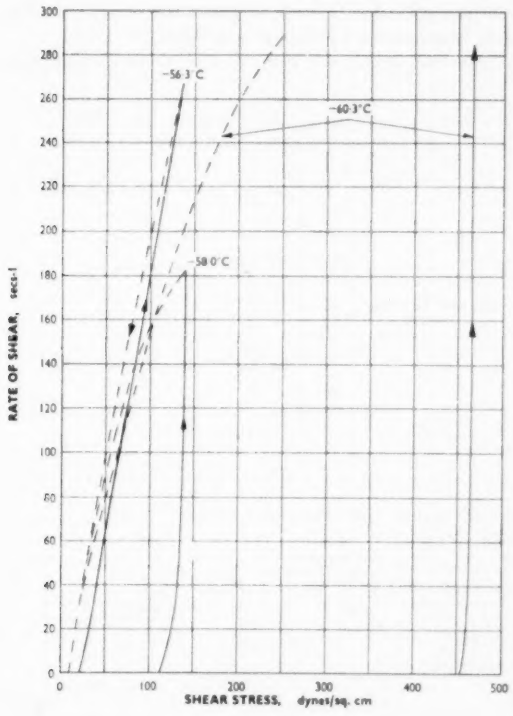

FIG 2

RHEOLOGICAL PROPERTIES OF REFERENCE KEROSINE SR 312
At three temperatures

Full-scale tests show that for flow upstream of the booster pump the pumpability temperature limit is set by failure of fuel to flow along the tank bottom towards the pump inlet. This normally occurs before failure of fuel to enter the pump, and although instances have been reported of blockage of the screens used in booster pump inlets, where these have been of unusually fine mesh, it is unlikely that such a fine mesh screen will in practice be found necessary.

There is less shear stress available to cause flow towards the booster pump as the level of fuel in the tank falls. Thus it is found that the criterion is not merely the ability of the pump to extract fuel from the tank, but its ability to continue to do so until all, or nearly all, of the fuel has been removed.

Current specifications for aviation fuels control low temperature properties by means of a freezing point clause (IP Cold Test, 16/44T, or ASTM Freezing Point D1477-57T), coupled with a restriction on viscosity at some moderately low temperature. However, the limit of pumpability from the tank (defined as the temperature at which virtually all of the contents can be evacuated) is found to bear little relation to either property. It occurs at a temperature below the freezing point, after the fuel has acquired a semi-solid nature, and the margin between freezing point and pumpability limit varies widely between fuels. It is usually in the range 3° to 15° C but cases have been encountered outside this range. The pumpability limit bears no relation to viscosity for fuels in the kerosine range.

As would be expected from the nature of the process, the IP Pour Point (IP 15/55) is found to give a better indication of pumpability limit. The margin between pour point and pumpability limit ranges roughly from zero to 8° C, a variation which is attributed largely to the imprecision of the test method.

No difficulty is experienced in causing the fuel to pass through the booster pump itself, since sufficient shear force is available to overcome the yield value down to temperatures well below the pour point.

The behaviour of cold fuel downstream of the booster pump depends markedly upon whether the reduction in temperatures occurs with the fuel flowing or static. If flowing, the high rates of shear to which the fuel is subjected by the booster pump are sufficient even at temperatures well below the pour point to break down the structure of the highly thixotropic semi-solid, so that the principal resistance to flow is imposed by the viscosity of the liquid phase. This will not be limiting under practical conditions. If the fuel is static when the pipe is cooled, however, the semi-solid formed by the separation of solid material below the freezing point can rapidly develop a sufficiently large yield value to exceed the relatively small shear stress which can be applied by the booster pump, especially if the pipe is a long one. This shear stress is of the same order as that produced within the fuel tank by gravitational forces, so that the same criterion of pumpability can probably be used.

Fuel Temperatures in the Aircraft

When establishing the fuel temperatures which can be experienced in flight, it is necessary to consider two factors: (*a*) the ambient temperatures encountered in the flight paths, and (*b*) the extent to which these temperatures are transmitted to the fuel. In addition to knowing the minimum ambient temperatures recorded in the areas of interest it is desirable to have some statistical data as to the frequency with which given temperatures occur, since the very lowest temperatures may occur extremely rarely. Suitable data, compiled by BOAC, were presented in an earlier publication [1] and are reproduced in Figs 3 and 4 for the Arctic and the tropics respectively. The curves indicate the temperatures exceeded on 85 per cent of occasions, whilst the absolute minimum temperatures

recorded at the various altitudes are shown as individual points. January temperatures in Goose Bay and Colombo were selected for the statistical treatment as representing the lowest for which records are available, and these results are thought to be representative of the worst conditions experienced in either hemisphere.

Two factors prevent the fuel temperatures falling to the ambient air temperature. First, the rate of heat transfer from the fuel is finite and influenced amongst

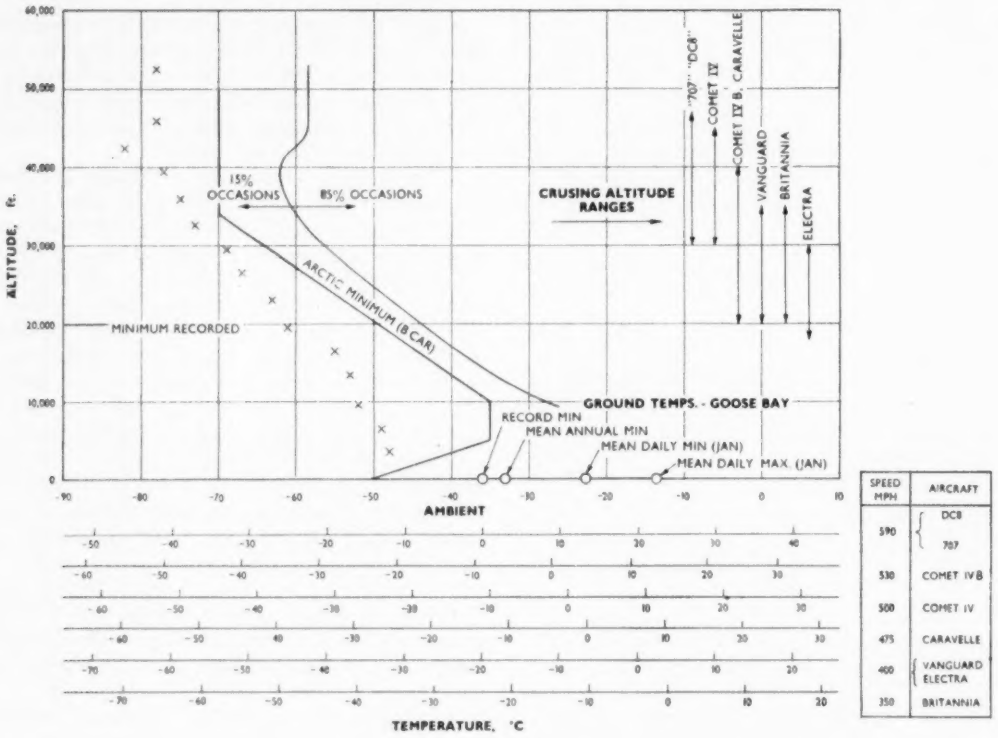

FIG 3

AMBIENT TEMPERATURES AT ALTITUDE IN ARCTIC AND AIRCRAFT TEMPERATURES CORRECTED FOR KINETIC HEATING

livery pipe in which flow has not yet commenced. It is therefore safer in general to assume that the fuel will reach the final equilibrium temperature of ambient plus kinetic temperature rise. Equilibrium temperatures can exceed the estimated value due to the proximity of heated portions of the aircraft—cabins, engines, de-icing air ducts, etc.—but these effects are best ignored until actual temperature measurements have been carefully recorded in flight.

Referring again to Figs 3 and 4, it is therefore

other things by the degree of insulation provided by the tank walls. Secondly, the equilibrium temperature approached by the fuel differs from the ambient value by the temperature rise due to kinetic heating, which is proportional to the square of the air speed. The first factor is significant on relatively short stage lengths, especially with bag-type tanks, which provide insulation. However, it is difficult to estimate its effect with accuracy, since heat transfer rates vary from one part of the fuel system to another, and the temperature of the fuel is not uniform. For example, the fuel in the middle of a large tank can be considerably warmer than that either at the outside, or in an interconnecting pipe between tanks, or in a fuel de-

possible to insert new abscissa scales, for the speeds appropriate to the various aircraft, which will indicate the equilibrium fuel temperatures. Using these scales in conjunction with the cruising altitude shown for the corresponding aircraft enables the temperatures which each are likely to encounter to be read off.

Table I shows these aircraft temperatures for the minimum recorded ambient temperature and for the temperature exceeded 85 per cent of the time, assuming flight at the maximum cruising altitude. It may be seen that the lowest temperatures either in the Arctic or tropics are predicted with the Britannia. This is due to the combination of a fairly high maximum cruising altitude with a moderate cruising speed

in this long-range propeller-turbine aircraft. (With jet aircraft the higher altitudes are more than counteracted by the greater kinetic heating resulting from the increased speed.)

stabilize at a value 6° or 7° C above the estimated value for an ambient temperature of the order of −70° C. This is presumably due to the incidental effects mentioned above. Thus, for the lowest re-

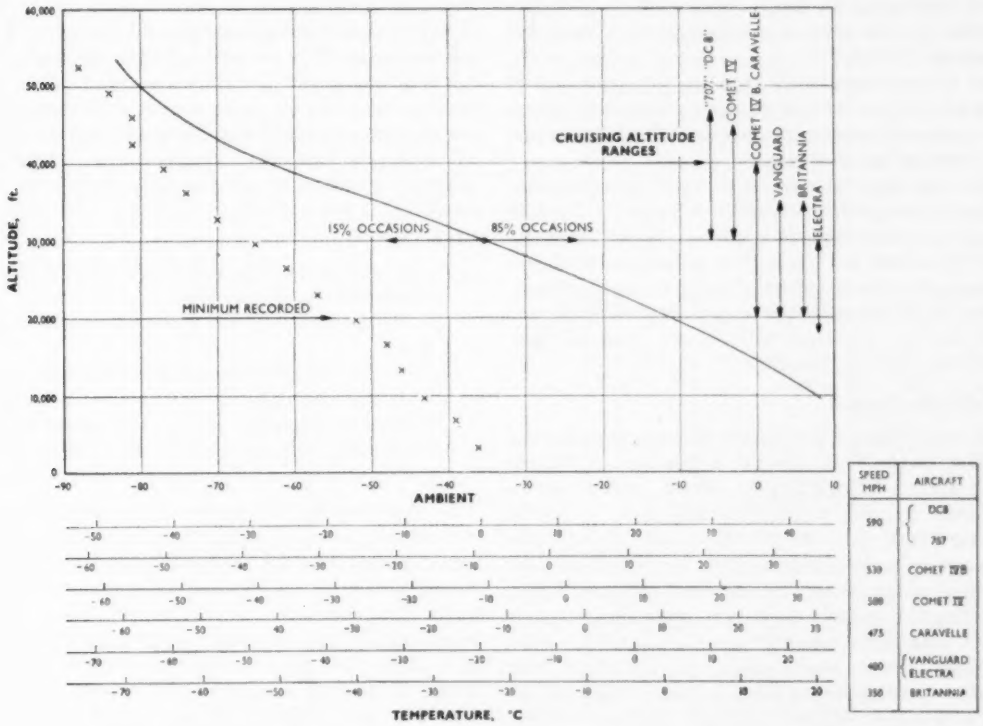

FIG 4

AMBIENT TEMPERATURES AT ALTITUDES IN TROPICS AND AIRCRAFT TEMPERATURES CORRECTED FOR KINETIC HEATING

It is understood that during extensive flight testing of the Britannia to obtain a direct measure of the fuel temperatures in various parts of the system, the minimum fuel temperatures have in fact been found to

TABLE I

Fuel Temperatures Predicted for Various Aircraft

Aircraft	Temperature exceeded on 85% occasions at maximum altitude, ° C		Minimum temperature at maximum altitude, ° C	
	Tropics	Arctic	Tropics	Arctic
Douglas DC.8 } Boeing 707 }	−41	−27	−47	−47
Comet IV	−48	−37	−55	−56
Comet IVB	−36	−35	−50	−50
Caravelle	−41	−40	−56	−55
Vanguard	−33	−44·5	−57	−58
Britannia *	−40	−48·5	−60	−62
Lockheed Electra	−20	−41·5	−50	−54

* Measurements in flight show these estimates to be some 6° or 7° C low.

corded ambients, the actual fuel temperatures in this aircraft will be in the region of −55° to −58° C, which are the values shown in the table for the Vanguard and the slower of the jet aircraft.

The pure jet aircraft give higher minimum temperatures, −56° C for the Comet IV −50° C for the Comet IVB, and above −50° C for the faster Douglas DC.8 and Boeing 707.

It should be emphasized, however, that these are the temperatures based upon minimum recorded values. Reference to the table for temperatures exceeded on 85 per cent of occasions shows that for most of the time these will be well above the minimum. The lowest fuel temperatures which have been quoted will therefore be met only on the very rare occasions when flight at maximum altitude coincides with the occurrence of minimum ambient temperatures for an appreciable proportion of the flight. Airlines generally accept the fact that they may need to make flight diversions or reductions in altitude in such cases of exceptionally low atmospheric temperature.

At the present time the airlines are expressing the low temperature properties of the fuel they require in terms of freezing point, and −50° C freezing point fuels are generally acceptable. However, they realize that freezing point is not a true criterion of pumpability, and many of the European airline operators have stated that their true requirement is for a fuel pumpable at −55° C.

The above discussion shows that this figure should cover all but extremely rare cases, and indeed actual flight measurements might show that this temperature will never, in fact, be reached.

On a long-term view, it seems that the present long-range propeller-turbine aircraft will represent the most difficult case to meet. It is already evident that the faster jet aircraft are less critical, as the greater kinetic heating outweighs the effect of their increased altitude. Indeed, with the advent of supersonic transport aircraft the fuel problems will become those of high, rather than low temperature.

Fuel Supply Position

For many years after the war aviation kerosine was supplied in the U.K. against a Ministry of Supply specification, DEngRD2482, which called for a maximum freezing point (IP Cold Test or ASTM freezing point) of −40° C. However, about three years ago, with the prediction that temperatures lower than this would be experienced, fuel suppliers began marketing turbine fuel meeting that specification, but with a maximum freezing point of −50° C. Subsequently, a new specification, DEngRD2494, was issued by the Ministry of Supply to cover this fuel for their long-range military transport aircraft. It has been found possible to supply such a fuel, at no increase in cost to the consumers, by the selection of material from suitable sources. It is probable that −50° C is the lowest freezing point which can be supplied in this way on a worldwide basis without introducing manufacturing problems and increasing the cost of the fuel. The fuel cost represents such a high proportion of the operating costs of turbine-powered aircraft that every effort must be made to avoid increasing it.

Possible Solutions

From the preceding discussion of the nature of the pumpability requirements, and the conclusion that the freezing point, although a safe criterion of pumpability, is an unnecessarily restrictive one, it is clear that the first measure to be taken in meeting the operational requirements is to devise and introduce a more realistic pumpability criterion for fuel specifications.

Since the IP pour point test measures the right sort of fuel property, and has been found in full-scale tests to give a closer indication of pumpability limit than does the freezing point, this test has, in the absence of anything better, been suggested as a replacement for, or complement to, the freezing point in a number of proposed specifications.[2]

However, the pour point test was not designed for use at such low temperatures as those in question; it was intended for use with heavier products, often subject to complicating thermal history effects which in any case militate against the achievement of a very precise result. For use with aviation kerosines, where the closeness of the operational pumpability requirement to the economic production limit demands a very precise test, and where the absence of thermal history effects means that there should be no fundamental difficulty in achieving good reproducibility, the pour point test is not sufficiently sensitive. Its main disadvantages are:

(1) the excessively wide steps of 5° F (3° C) in which the test is conducted;

(2) the necessity for subjective assessment of flow;

(3) the repeated removal of the test sample from the cooling bath, which prevents the maintenance of a uniform sample temperature. This third point also lessens any chance of improving (1) by reducing the temperature increments.

In searching for an improved pumpability test, it was at first thought that since the controlling rheological property of the fuel is its yield value, a direct determination of this value would constitute the best test. This was done, as mentioned earlier, by determining the pressure required to force the fuel out of a small-bore glass U-tube, in which it had been cooled to a given temperature. However, although with care this method gave repeatable results using some fuels, which agreed with the results obtained in the conicylindrical viscometer, considerable difficulties arose with other fuels, and with these good repeatability was not achieved. These difficulties appeared to be associated with the occurrence of abnormally low flow rates, and of cases where the flow would stop after proceeding for a short time at a given pressure. Similar effects were also observed in the conicylindrical viscometer. It was thought that the phenomena were associated with the small scale of the equipment—tubes of 0·5 and 1·5 mm were generally used, and the radial clearance of the viscometer was 0·3 mm—so in view of these results, and the known difficulties which can be introduced by wall effects in small-scale systems, it was decided that a test carried out on a larger scale would be preferable.

Thus a new test has been devised which, it is hoped, should be suitable for control laboratory use. The test does not set out to measure any fundamental rheological property, although an attempt has been made to subject the fuel to conditions of shear stress similar to those experienced while flowing within a fuel tank towards the booster pump. It may be regarded as an improved pour point test, in which

steps have been taken to eliminate the disadvantages listed above.

Fig 5 shows a diagram of the apparatus, which comprises two cylindrical chambers connected by a downward-opening, spring-loaded poppet valve in the base

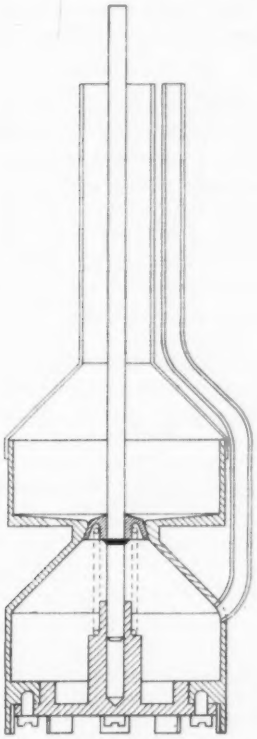

FIG 5
COLD FLOW TEST INSTRUMENT

of the upper chamber. The diameter of the valve is one quarter of that of the cylinder. 100 ml of fuel are introduced into the upper chamber, and the apparatus immersed in a low temperature bath at a closely controlled temperature for a sufficient time for the fuel temperature to reach that of the bath. The poppet valve is then opened for a controlled period, after which it is closed again, and the apparatus removed from the bath. The volume of fuel remaining in the upper container is measured after the apparatus has warmed up, the amount being an indication of pumpability characteristics.

The apparatus differs fundamentally from a viscometer, which it might in some respect resemble, owing to the large size of the orifice. This means that most of the flow takes place as soon as the valve opens, and the result obtained depends primarily upon the rigidity of the semi-solid fuel, or yield value, and to only a minor degree upon the flow rate, or apparent viscosity. However, in practice the extent to which fuel can be pumped from a tank will depend to some small extent upon the apparent viscosity at the flow rate required, and the apparatus can be made responsive to this by restricting the opening period of the valve to a suitable value.

The method has been evaluated on a range of different fuels to determine the nature and repeatability of the results, and to compare them with the results of other laboratory determinations. Fig 6 shows results obtained on a number of fuels, plotted as "percentage fuel remaining in upper container" or "percentage hold-up" against temperature. The freezing point and pour point of each fuel is also shown. All the curves are fairly steep, and may conveniently be represented by straight lines in the middle

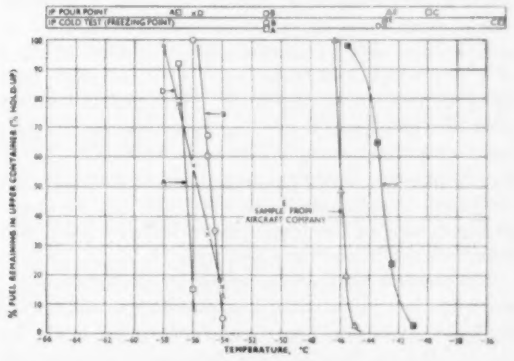
IP POUR POINT
IP COLD TEST (FREEZING POINT)
% FUEL REMAINING IN UPPER CONTAINER (= HOLD-UP)
SAMPLE FROM AIRCRAFT COMPANY
TEMPERATURE, °C

FIG 6
TYPICAL RESULTS OF COLD FLOW TEST SHOWING IP COLD TEST (FREEZING POINT) AND IP POUR POINT OF FUELS TESTED

section. Repeatability is very good, normally better than $\frac{1}{2}$° C, and appears to depend entirely upon the accuracy of bath temperature control.

Since it is thought that the viscosity of present fuels is sufficiently low to have only a small influence upon pumpability limit, a valve opening time of 10 seconds has been used, as it is found that, for the fuels tested, most of the flow which will occur has taken place in this time.

The choice of valve opening time, and of the "percentage hold-up" value which is taken to represent the pumpability limit, can only be finally decided by correlating tests in this apparatus with tests in actual aircraft installations. However, in view of the steepness of the curves, the selection of a suitable value for percentage hold-up should not be very critical. Experience to date suggests that 30 per cent hold-up would represent a reasonable indication of pumpability limit. This might seem to be a high value to be considered acceptable, but it must be remembered that the much greater surface/volume ratio in the apparatus than in an aircraft increases greatly the severity of the test.

The test method requires the use of a closely controlled temperature bath, but it is felt that this will be necessary for any precise and repeatable test which might be devised. By circulating alcohol from the bath through cooling coils in another vessel containing solid CO_2 and alcohol, and trimming the temperature by means of a thermostatically controlled heater, it has been found possible to maintain a constant temperature within $\pm 0{\cdot}2°$ C. If used as a control test it is envisaged that it would be carried out at only one specified temperature, at which a certain maximum percentage hold-up would also be specified. The test could therefore be of a "pass" or "fail" type.

Results from different laboratories suggest that the reproducibility of the method is good—within 1° C—and steps are being taken to obtain a correlation with the fuel performance in aircraft systems. Although results from tests of practical systems can only be accumulated slowly, an example is included in Fig 6 of a fuel which had been tested in a full-scale tank rig by an aircraft manufacturer. This fuel, laboratory tests of which gave a freezing point and an IP pour point separated by only $\frac{1}{2}°$ C at approximately −43° C, was in practice found to be pumpable from the aircraft tank at an adequate rate at −46° C. As shown in Fig 6, this is the temperature which would have been forecast in the proposed test.

The method has been accepted by the IP as one of several to be evaluated for its suitability as a Standard Method. It is also being put forward for study by the ASTM Low Temperature Pumpability Group. It is at present referred to as the Thornton Cold Flow Test.

Using this pumpability criterion in place of the freezing point, it is found in the author's group of companies that in general the fuels produced to a −50° C freezing point specification do in fact satisfy the requirement of pumpability at −55° C. Thus it seems probable that merely by the use of this or some other suitable pumpability test, it would be possible to meet the operational requirements satisfactorily.

However, in the unlikely event of future practical experience showing a demand for a pumpability limit somewhat below −55° C, or if the supply position should change for any reason, other measures will be necessary.

Treatments which can be applied to improve the low temperature properties of a kerosine are either costly or may restrict availability of the product.

The use of an additive to improve pumpability would be attractive, especially for dealing with individual batches. In the past little or no success has been achieved with conventional pour point depressants in kerosines, although these can be highly effective in much heavier material. This must, however, be attributed largely to the insensitivity of the pour point test, and its unsuitability for research work, as emphasized in the paper by Hutton,[3] since the author has recently found appreciable reductions in pumpability limit using the Cold Flow Test. For instance, in one kerosine 1 per cent of a proprietary pour point depressant reduced the pumpability limit by well over 15° C, and at 0·1 per cent concentration the reduction was 6° C.

Fig 7 shows the results obtained with several proprietary additives used at two different concentrations

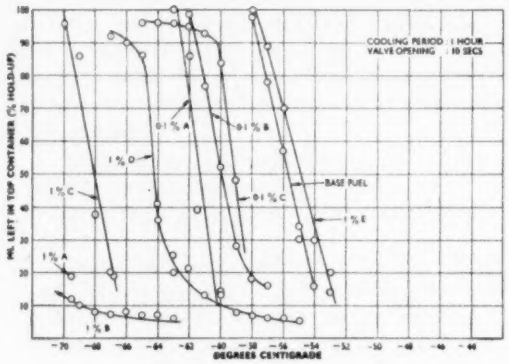

FIG 7

KEROSINE "1" WITH VARIOUS PROPRIETARY POUR POINT DEPRESSANTS

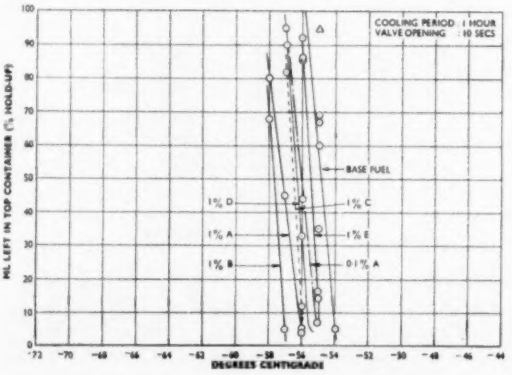

FIG 8

KEROSINE "2" WITH VARIOUS PROPRIETARY POUR POINT DEPRESSANTS

in this kerosine. It is clear that they vary widely in effectiveness.

In Fig 8 the effectiveness of the same additives in a different kerosine is seen to be very much less. This illustrates the selective nature of the additives' action, and emphasizes the need for taking into account variations in fuel constitution when dealing with such additives.

These results are quoted to show that the additive approach has some promise. It would clearly be necessary to produce additives which are either cheaper, or effective in lower concentrations than the

conventional ones examined, but since these were developed specifically for very different materials the possibility of producing suitable additives for use in kerosines should not be discounted. The paper by Hutton [3] shows that various possible mechanisms exist by which these might be effective.

CONCLUSIONS

1. The low temperature limit of fuel performance in a civil turbine-powered aircraft equipped with fuel line heaters is generally imposed by failure of the fuel to flow within the aircraft tanks, due to the commencement of solidification. If fuel has cooled statically in part of the delivery pipe system, inability to start flow will also occur at very nearly the same temperature. The controlling fuel property is neither the freezing point (IP Cold Test) nor the viscosity, but the yield value which the semi-solid fuel develops.

2. Under the very worst combination of flight procedure and atmospheric conditions, the fuel temperature might fall to, or very slightly below, −55° C. This temperature can be taken as the lowest requirement, since if lower temperatures should ever be met, they will occur so rarely that flight diversions or reduction of altitude would be acceptable.

The lowest temperatures occur with long-range propeller-turbine airliners. Higher speed jet aircraft are less critical.

3. Fuels at present being supplied against a freezing point specification of −50° C are in general pumpable at −55° C, but it would be far more satisfactory to specify a criterion of pumpability instead of a freezing point.

4. No existing control test provides a suitable criterion of pumpability. A new test has been proposed which should be satisfactory for control use, and which has been shown to possess good repeatability and reproducibility.

5. Although pumpability requirements can at present be met, supply difficulties could arise in the future, so that measures to improve low temperature performance may have to be taken, *e.g.* by the use of additives.

ACKNOWLEDGMENT

The author wishes to express his thanks to the directors of " Shell " Research Ltd for permission to publish this paper.

References

[1] Strawson, H. *Shell Aviat. News*, 1955, (210).
[2] Proposed specifications by: (1) ASTM; (2) American Airlines; (3) Trans-World Airlines; and (4) Society of British Aircraft Constructors.
[3] Hutton, J. F. *J. Inst. Petrol.*, 1959, **45**, 123.

DISCUSSION

J. R. Lodwick: The subject of low temperature characteristics of middle distillates has assumed increasing importance of late in connexion with the service behaviour of diesel and jet fuels. For my own part I accept, with some reservations, which I will enlarge on later, that the conventional methods of assessing low temperature behaviour are not all they might be, and I am sure that all present will be gratified to know that a Working Group of the General Test Panel of IP Sub-Committee No. 3 has been set up to examine the repeatability and reproducibility of the test methods proposed for assessing the low temperature pumpability of aviation turbine fuels.

The three methods I know of, which have been proposed to date (and there may be more), are the Thornton Cold Flow Test, the Esso Coni-cylindrical Viscometer Test, and the British Petroleum Laboratory Pumpability Test. I would like to stress the necessity of having available a reliable and generally accepted standard method of test for the low temperature pumpability of aviation turbine fuels and I hope that the working group mentioned will make their deliberations with all possible speed.

There are a few points in both papers on which I should like clarification or additional information.

Mr Strawson states that the margin between the IP Cold Test and the pumpability limit is in the range 3° to 15° C, and is sometimes outside this. Later he states that this margin for the pour point is from zero to 8° C, a variation which is attributed largely to the poor precision of the test method. I would say that these wide deviations are not apparent in the experimental data given in Fig 6 and, further, it has been found at the BP Research Centre that over a wide range of kerosine hydrocarbon type composition that the deviation between Cold Test and pumpability limit as assessed on both the pressure and suction sides of a pump in a rig test simulating an aircraft fuel system varies from 2° to 8° C, while the corresponding deviation for a modified (1° F steps) pour point varied from 0° to 4° C. I should like to know whether the fuels giving the very wide deviations quoted were unusual in character and I also observe, in some contradiction to the statement of Mr Strawson's paper, that the use of smaller increments in the pour point test appears to have had some beneficial effect. The statement made that fuels at present being supplied against a cold test specification of −50° C are in general pumpable at −55° C, with which most workers in the field agree, also seems to suggest that a range of 3° to 15° C in the cold test/pumpability margin is rather wide, at least for representative commercial fuels.

The use of the flow test for assessing the usefulness of pour point depressants is of considerable interest, and in view of the optimistic note sounded in the papers, I should like to ask whether the use of pour point depressants is free from all undesirable side effects in jet fuels.

I note that Mr Hutton states that fuel filters can be blocked with waxy materials at temperatures well above the measured cloud point. I find this observation difficult to accept, both from experience involving density measurements and from the work of Dr Ruf, head of the Swiss Federal Material Testing Laboratory, who, as a

result of extensive diesel fuel field and rig testing, concluded that the cloud point was certainly not a bad criterion of the tendency of paraffinic fluids to obstruct filters, but the appreciation of other types of gas oils on this basis might in some cases be too severe. No filter clogging above the cloud point was experienced. Has Mr Hutton any observations to make on this point?

My last question concerns the Einstein equation quoted by Mr Hutton. I feel that it would have been useful to emphasize that where wax/oil systems are being studied and it is desired to apply the Einstein relationship for the viscosity of a suspension, then the volume concentration term in that relationship should be calculated on the basis of the total volume of the fragments, including the trapped oil.

If, for example, the work reported by Bondi [4] in Mr Hutton's paper is analysed it will be seen that the C factors calculated for 5·0 per cent (not 0·5 per cent as quoted by Bondi) paraffin wax or ozokerite in a medicinal white oil were 0·45 and 0·91 respectively. This can be interpreted as meaning that some 40 per cent of oil in the first case and 86 per cent in the second case is trapped or adsorbed by the wax particles, which seems to bring the wax/oil system more in line with soap/oil systems with respect to the magnitude of the true C factor. This conclusion is reached by applying a modified equation to the data where C is equal to $\frac{W + Ao}{100}$, where W is the per cent volume of wax particles and Ao the per cent volume of the trapped or adsorbed hydrocarbon oil.

H. Strawson: I should first like to agree fully with Mr Lodwick about the desirability of reaching an agreement as soon as possible on a standard test for pumpability, whichever method is finally chosen.

Regarding the margins between cold test and pumpability limit quoted in the report, the fuel giving a margin of 15° C was in fact a standard aviation turbine fuel, although it was not one with a cold test point as low as −50° C.

The fact that I said later in the paper that the fuels at present being supplied to a cold test point specification of −50° C are found to be pumpable at −55° C should not be taken to infer that these fuels all necessarily show a margin of 5° C. Some have a smaller margin, but their cold test values are considerably below the specification maximum for cold test.

J. F. Hutton: I must first of all explain that the problem of flow of fuels at low temperatures is relatively new ground to me. Consequently my paper is theoretical and no doubt idealistic in outlook. Nevertheless, it seems to me that a return to first principles was not a bad way of trying to sort out the confusion that exists in the published literature.

Mr Lodwick has raised two specific points. First, the question of whether or not filters block with waxy material at temperatures above the measured cloud point. My practical experience with the problem of filter blocking is nil. However, my colleagues at Thornton who have had practical experience of it have informed me that blocking at temperatures above the cloud point does occur. The occurrence is rare and appears to be confined to vehicles which, with full fuel tanks, have been allowed to stand idle for a long period during which the ambient temperature has fallen for a while to a level below the cloud point. The fact that in the wide experience of Mr Lodwick and Dr Ruf (whose papers I have not seen) the effect has not been observed confirms its rarity.

Secondly, the Einstein equation. The original equation is quoted in the paper to illustrate the sort of relationship that will apply to suspension of wax in oil when the structure is completely broken down. Mr Lodwick is right to point out that it cannot be expected to apply without special restrictions. There are many papers and books on rheology explaining the restrictions and setting out alternative equations.

Concerning the numerical examples taken from Bondi's paper, I shall say that the conception of wax particles with trapped or associated oil giving an effective increase in C is only one interpretation of the data. This picture becomes unsatisfactory when experimental data, as they can do, lead to a value of the volume of trapped oil greater than 100 per cent. A more usual interpretation is that used by, *e.g.*, Bondi himself, *i.e.* the factor 2·5, which applies only for very dilute suspensions of non-interacting rigid spheres, is increased by both the irregular shape of the wax particles and the interaction to form a structure.

When the structure is destroyed by shearing or perhaps by the addition of pour point depressants, the numerical factor will give a measure of the shape of the wax particles.

A. G. C. Fairman: I found Mr Strawson's paper particularly interesting, since many of the statements made confirm the conclusions which have been drawn from

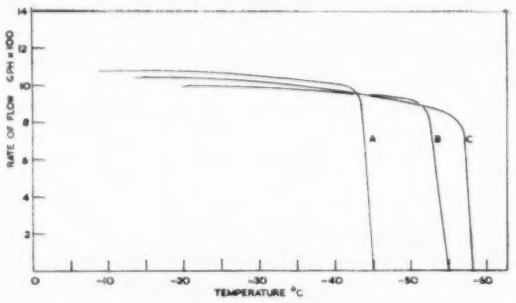

FIG A

work that has been going on at the BP Research Centre. Since the data presented are generally along the lines of our own investigations, it might be helpful if I tried to add a little to the general picture. Fig A shows a series of three flow *v.* temperature curves obtained on three different kerosines, A, B, and C, which have nominal cold test values of −40°, −50°, and −55° C. It will be seen that over quite a wide temperature range the flow rate falls off very little indeed until the temperature is within two or three degrees of the limiting value for pumpability, when the flow falls to zero. These tests were carried out in a pumpability rig which was designed to simulate an aircraft fuel system and to measure the conditions on the suction side of the booster pump. These tests were, we found, very repeatable indeed, the limiting temperature for a particular kerosine occurring repeatedly within a very narrow band.

In some aircraft we believe the limiting conditions to be on the suction side of the pump, while in others it is on the pressure side of the pump. Here we are taking the condition of the kerosine on the pressure side of the pump as the limiting criterion. What we attempted to do was to simulate an aircraft ascending from ground level to altitude, and this we did by dropping warm kerosine into a pre-cooled tank and then progressively cooling it. The lower line (Fig B) shows the ambient

temperature in the chamber; the top solid line shows the temperature of the kerosine in the tank, which, as can be seen, was decreasing relatively slowly. The dotted line shows the temperature of the kerosine in the coil, which was connected to the booster pump and to a suitable means of measuring flow rate outside the cooling chamber.

In some aircraft a flight plan is followed in which alternate tanks are used. This means that on occasions there are lines containing static kerosine and this is what we have been simulating. It can be seen that when the kerosine in the line is static, its temperature falls fairly rapidly towards the ambient temperature. In fact, this is Kerosine A, shown in Fig A, which had a pumpability limit of about $-46\frac{1}{2}^\circ$ C. It can be seen that the temperature of the static kerosine is falling fairly rapidly towards that value.

I think the lesson to be learned here is that by a modification to a flight plan, one could extend the time to failure. This could be done by shortening the pumping and rest periods so that the rate at which the line temperature falls towards the critical value is retarded. It might be of interest that the saw-tooth effect, shown in Fig B, has been experienced in flight trials.

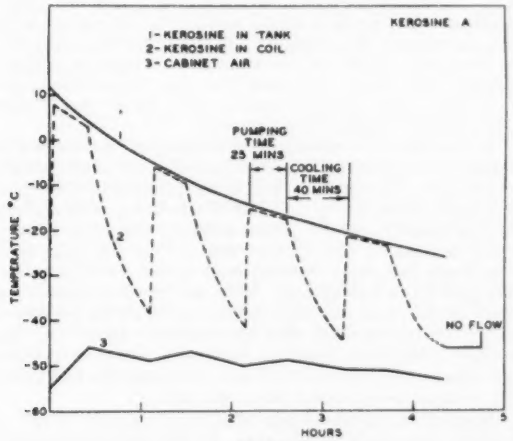

Fig B

SIMULATION OF AIRCRAFT FLIGHT PLAN

In our work we have not forgotten the important task of correlating large-scale rig data with laboratory tests, because it is obviously impossible to carry out a full-scale test every time an assessment on a kerosine is required. We have tried to correlate our rig data with typical laboratory tests. These have included the pour point, cloud point, cold test, and setting point methods. We have found surprisingly good correlations with all of them, with the cold test always giving values rather higher than the pumpability limit as set in the rig; the cloud and pour point methods giving a scatter about the rig results; and the obsolete setting point method giving results below the rig results. Fig C shows pour point results against rig results, because both Mr Strawson and Mr Hutton mentioned the pour point in their papers. I should mention that this was a modified pour point method, in that we examined each sample at smaller increments of temperature than the normal 5° F, so that this possibility presents a rather better picture than one would obtain if one examined a sample every 5° F.

I do feel that there is a better laboratory method than the pour point for predicting pumpability limits, as has been mentioned earlier. The method I refer to gives a much better correlation with rig results than the pour point method. However, I do not propose to say more

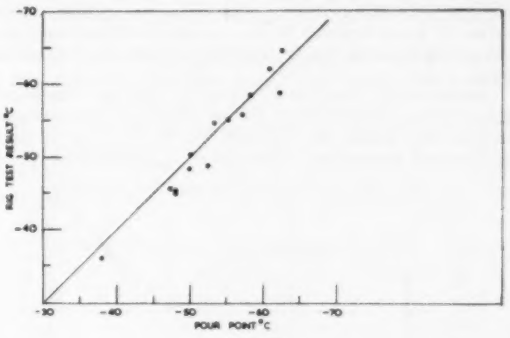

Fig C

about it at this stage because I feel that considerations of the various test methods should be the responsibility of the IP working group which has been set up to study them.

B. T. Fowler: As the previous speaker has just intimated, rig results are an essential preliminary step to devising a laboratory prediction test for fuel pumpability.

We have adopted this practical approach to the problem of low temperature pumpability of jet fuels and I would like to put the results of this work before you now.

Fig D is a simplified diagram of our aircraft fuel system rig. Its existence owes much to the advice and loan of equipment by companies of the U.K. aircraft industry. Dots represent thermocouples recording fuel temperature

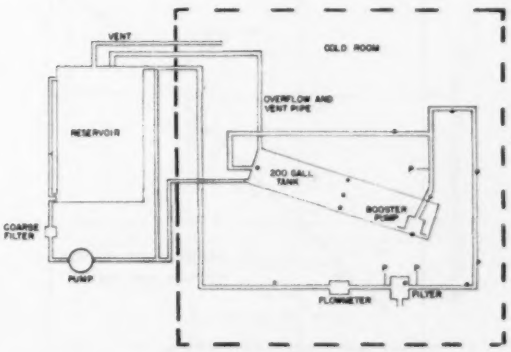

Fig D

LOW TEMPERATURE PUMPABILITY RIG

throughout the system and P's pressure gauges. Pipe plumbing and insulation was so arranged that booster pump suction and pressure side failures could be investigated. In addition, failure due to wax blockage of the fuel filter was also studied.

In all, nine fuels of widely different origin have been tested in this rig, with the following results.

First of all, filter failure due to wax blockage could be predicted by the IP cold test. However, it is now generally agreed that this criterion of failure is no longer

M

applicable, as most commercial turbine-powered aircraft incorporate a fuel heater upstream of the filter to avoid problems due to icing.

Suction and pressure side failures of the booster pump were found to occur within 2°–3° F of each other, with suction side failure being slightly more critical. Both types of booster pump failure occurred at temperatures appreciably below the IP freezing point of the fuel under test.

Appreciating that fuel viscosity was an important factor influencing fuel flow laboratory viscosity, measurements were made on the test rig fuels using a conicylindrical viscometer, this type of instrument being

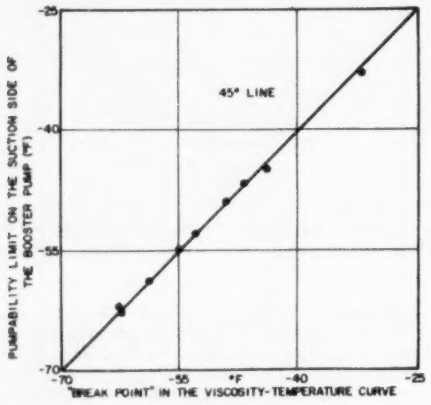

FIG E

CORRELATION OF PUMPABILITY LIMIT ON THE SUCTION SIDE OF THE BOOSTER PUMP AND "BREAK POINT" OF THE VISCOSITY–TEMPERATURE CURVE

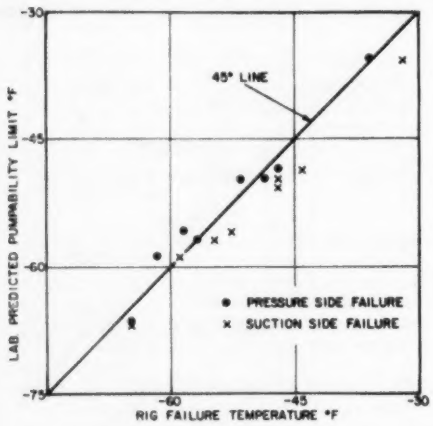

FIG F

LABORATORY PREDICTED FAILURE *v.* RIG FAILURE

particularly suitable for viscosity measurements on non-Newtonian fluids.

It was noted that as the temperature of the fuel was decreased, viscosity gradually increased until a point was reached where a further small decrease in temperature resulted in a very large increase in fuel viscosity.

As illustrated here (Fig E), the break point in the viscosity curve gave excellent correlation with suction side booster pump failure in our cold room test rig.

Whilst not anticipating the deliberations of the IP and ASTM panels formed to standardize a pumpability test method, we would point out that, although the instrument we have used gives a very precise answer, it is rather an expensive piece of equipment for a routine test laboratory.

We have therefore compared our rig results with answers obtained by a much simpler and cheaper laboratory test method developed by BP and correlating with their own fuel system rig pressure side failures. I feel sure that more will be heard of this very promising test method, and I will therefore finally show the correlation obtained between their laboratory test and our own rig results (Fig F). The correlation between laboratory and rig results is very promising.

H. Strawson: May I reply to the last two speakers, first agreeing with their remarks about the function of the IP working group. It is clearly up to this group to select the best test, and this should be one which both correlates with the aircraft requirements and is convenient for use in a control laboratory.

The curves which Mr Fairman showed of flow rate against temperature were very interesting in demonstrating that the rate does not fall off appreciably until the limiting temperature has been nearly reached. This confirms what we have found, and for this reason we have not considered the viscosity, or the apparent viscosity, in the early stages of thickening as a very important factor. We have concluded that the important questions are "Will the fuel flow from the tank?" and "Can the tank be completely emptied?"

Both speakers mentioned the freezing of fuel whilst it is static in the pipes. We certainly recognize this as a very important consideration, and I am pleased to hear that they each find, as we do, that the limiting temperature for pumpability on the pressure side of the pump is very close to that on the suction side. This has also been confirmed in a paper recently read before the SAE on the Douglas DC.8 fuel system. This described tests showing that downstream blockage did not occur at temperatures down to the suction side pumpability limit, *i.e.* the booster pump can build up sufficient pressure to force solidified fuel out of the line, provided that the fuel is not too cold to be extracted from the tank.

Regarding the possibility of persuading the airlines to modify their flight techniques, by shortening the times between the use of different tanks, I feel one would have to be careful about imposing limitations of that sort. It could be difficult to guarantee that the air crew always followed such a procedure. Therefore I think that we have to be sure that the fuel will flow down the lines adequately without calling for special operating procedures.

Finally, Mr Fairman's slide showing the correlation between pour point and pumpability limit. This appears to be quite good using the modified method, but I noticed that in fact the points vary from something like 5° on one side of the line to 2° on the other. Admittedly this is closer than the variation I have observed, possibly using a wider range of fuels, but in any case I do not think that the agreement could be regarded as sufficiently good for a control test. We must still look for a better test.

J. F. Hutton: The failure on the suction side of the booster pump is presumably caused by what a grease technologist calls cavitation, *i.e.* the pump delivers air instead of fuel owing to the failure of the gelled fuel in the tank to flow under its own weight to the pump intake. Cavitation is a yield stress phenomenon. Therefore one would expect a correlation between the pump failure

temperature and the temperature at which the yield stress reached a certain critical value.

However, we have been shown a 1 : 1 correlation between the temperature at which failure occurs on the suction side and the break point in the viscosity/temperature curve.

Whether or not these two correlations are identical depends on the material and the test procedure. For a material which on cooling changes from a liquid to a mechanically stable gel the correlations should be identical. This follows because the apparent viscosity, the quantity usually measured with a coni-cylindical viscometer, is dependent on the yield stress. However, from the literature we find that the structure that imparts a yield stress to a gelled oil is broken down by continuous shear or by stirring lubricating oils [3, 5] and aviation fuels. [10] Furthermore, the equilibrium viscosity, reached after a period of shearing, is very close to the value of the viscosity of a wax-free oil and is not dependent on the yield stress of the undisturbed gel.

Therefore, I should like to ask: (1) whether the coni-cylindrical viscometer used by Mr Fowler was operated at very low rates of shear to specifically measure a yield stress, or at high rates of shear to measure an apparent viscosity, and (2) if the tests were carried out at high rates of shear, whether the value of apparent viscosity was measured as soon as possible after the start of the experiment, or after a long time when equilibrium has been reached?

L. B. Day: The viscometer used was one that could be operated at various shearing rates. We found that the position of the break point in the viscosity characteristic was independent of shearing rate over the range 40 to 1800 sec^{-1}. The technique for finding this temperature was to cool the fuel very slowly whilst continually shearing it. Thus the viscosity measured would be an equilibrium value. It is possible that the break point indicates the temperature at which the solid hydrocarbons precipitated are in sufficient quantity to form a structure, and if this be the case, then it is to be expected that fuel cooled in a tank to this temperature would be unable to flow readily to the pump.

S. C. Dodson: I should like to make a comment regarding the discussion of the use of the pour point to predict low temperature performance of A.T.K. A graph of results obtained at Sunbury has been presented showing a surprisingly good correlation between pour point determinations and rig tests, but we are not of the opinion that the test is entirely satisfactory. The fourteen points which were shown did not deviate from the fitted line by more than 4° C, but it must be remembered that these tests were made with smaller temperature increments than in the IP method.

In Fig G the results of suction side rig tests are shown plotted. One curve was obtained with a highly paraffinic fuel and the other with a naphthenic blend. With both fuels the flow fell off sharply as the limiting temperature was approached, with the paraffinic fuel showing the sharper decrease in flow rate.

In Fig H the effect of the head of fuel above the pump is shown. In the flow curves which have been shown by Mr Fairman and myself, the head of fuel above the pump was 14 inches. This depth was necessary to provide a sufficient volume for accurate measurement of flow rate, since the available direct reading flow meters were not found to be reliable under the conditions of test. However, it was felt that with smaller heads of fuel than 14 inches flow problems would be more acute, and so tests were made with a range of fuel depths. In these tests flow rate was not determined, but the temperature was noted at which the fuel ceased to be pumpable. If the limiting temperature at zero head is estimated from the graph it will be seen that it is only of the order of one degree different from that determined with 14 inches head.

Fig I is an interesting photograph of "solid" material in the tank. This photograph was taken after a suction

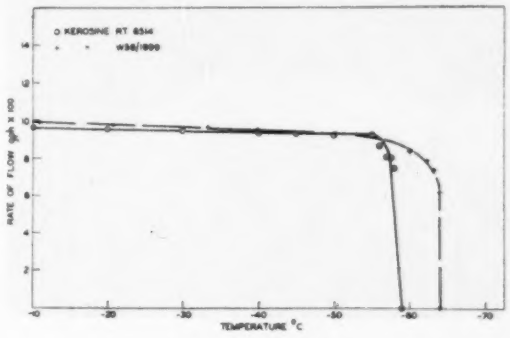

FIG G

RATE OF FLOW *v.* TEMPERATURE

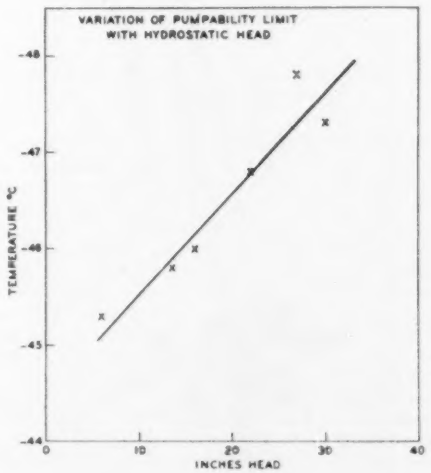

FIG H

KEROSINE BT 5926

side test. It should be remembered that these tests take a great deal longer than tests on the pressure side, which explains why so much kerosine has solidified. It can be seen that at the prevailing ambient temperature of 0° C the material was falling from the tank wall, and this occurred within three minutes of terminating the test. It seems from this test that if an aircraft reduced altitude when "solidification" occurred in the tank the "solid" material could be put into circulation, since it was found that these pieces would go into the pump when it was protected by a 10-mesh screen.

We were very interested to know the rate at which the "solid" kerosine was built up on the tank wall, and so a probe was designed (Fig J) with which the thickness

build-up could be measured without disturbing the tank. The instrument consisted essentially of a number of capillary tubes terminating at intervals of ½ inch from the bottom of the tank. A small air pressure was applied at intervals to each capillary tube and the time for blockage at each level was noted. The thickness of "solid" material was then plotted against time. It was

FIG I

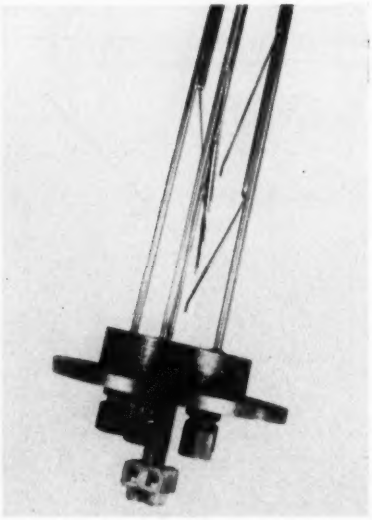

FIG J

found with the fuels tested that there was an initial delay period during which the fuel, which was initially at 10° C, cooled without reaching the "solidification" temperature. Eventually the tank wall temperature was reduced to the limiting temperature for the fuel under test, and following this the rate of build-up was observed to be constant.

For these tests a second probe consisting of a stack of thermocouples was mounted in the tank and the temperature was noted at each level when "solidification" occurred. These temperatures were, for any one kerosine, in good agreement with the temperature at which blockage occurred at the pump inlet and in the delivery line from the pump. Thus the available evidence indicates that the temperature at which flow ceases is not greatly dependent on the part of the fuel system which is investigated. Also it appears that fuel can be "lost" by adhering to a tank wall as well as to the bottom.

H. Strawson: Mr Dodson's results are most interesting. The tests with different fuel levels draw attention to a point which I have mentioned already but should like to enlarge upon somewhat, namely, the extent to which the tank can be completely evacuated.

In our full-scale tank tests, which were carried out with an aircraft tank, in a cold room, we removed the top of the tank to observe the behaviour of the fuel and were struck by the fact that at a temperature at which the booster pump could extract fuel readily when it was first switched on, flow towards the pump inlet would eventually diminish to such an extent that the pump began drawing in air, even though the more remote parts of the tank contained an appreciable quantity of partially thickened fuel. We therefore took this as a limiting case for pumpability and said that the pumpability limit would be the temperature at which a certain proportion of the tank contents, say 95 per cent, could be extracted before air entered the pump.

I am not quite sure that the extrapolation to zero of the pumpability limits measured by Mr Dodson for various heads of fuel automatically gives the temperature at which all the fuel can be pumped out. If a test is commenced at a given fuel level and continued until the head at the pump inlet is zero, a slope on the fuel surface caused by the thickening of the fuel can permit fuel to remain elsewhere in the tank.

I was interested in the theory that fuel which has solidified on the tank walls can be recovered when the aircraft descends into warmer air. I suggested this some time ago, but the idea was not taken up with much enthusiasm by aircraft operators! In fact, I believe that it does represent a definite safety margin, but one which is difficult to assess with any degree of accuracy and is therefore best disregarded for the present. Although large lumps of partially solidified fuel are readily digested by a booster pump, they can be easily held up by baffles or other distractions inside tanks.

On the question of there being one unique temperature for a given fuel, which controls all pumpability processes, I think that this is very roughly true, because a curve of yield value (or a related factor such as "hold-up" in our apparatus) against temperature is fairly steep. This is a useful fact when it comes to closing a laboratory test, since the critical yield value need not be known very precisely. However, there are fuels which show appreciably greater slopes than others and, if additives are used, the rate of change of these properties with temperature can be drastically reduced. Therefore one must guard against accepting too literally the idea that there is a unique critical temperature for any fuel.

A. F. D. Pott: The first of the two papers that we have heard is largely concerned with the theoretical aspects of flow properties of distillates at low temperatures, and as such provides an excellent framework from which we can try to explain the phenomena that occur in practice. For, as in so many things, the practical operation of diesel-engined equipment at low temperatures is not easy to reproduce in the laboratory and still less on paper. For example, the heat transfer from the engine to the fuel system and from the fuel system to its surroundings are features that vary enormously from vehicle to vehicle and are not reproducible in the laboratory. Last winter Esso Research Ltd carried out tests in the

Bavarian Alps and in N. Sweden, tests which covered a wide range of vehicles from agricultural tractors to heavy trucks under a wide range of temperatures and using fuels of most of the types likely to be encountered in Europe. Now tests of this sort, relying as they do on accurate predictions of weather, are frustrating and expensive, but nevertheless we think they are essential in order to tie up theoretical considerations such as we have heard with practical operation in the field. The criterion of satisfactory operation used in these tests was the ability to start the vehicle and to drive it for at least 30 minutes. The first fact to emerge clearly from our work has been that vehicle systems exhibited a wide variation of behaviour, some being far more critical than others. Fig K shows how the failure temperatures of eighteen vehicles were located in terms of the cloud points and the pour point of the fuels used. In fact, some vehicle fuel systems blocked when they operated at a temperature corresponding to the cloud point of the fuel used, whereas other systems operated satisfactorily down to the pour point. We found no examples of systems operating below the pour point, nor failing above the cloud point. The blockage invariably occurred at some point in the fuel system before the primary pump—the fine filter interposed between the primary pump and the injection pump did not block, presumably because by

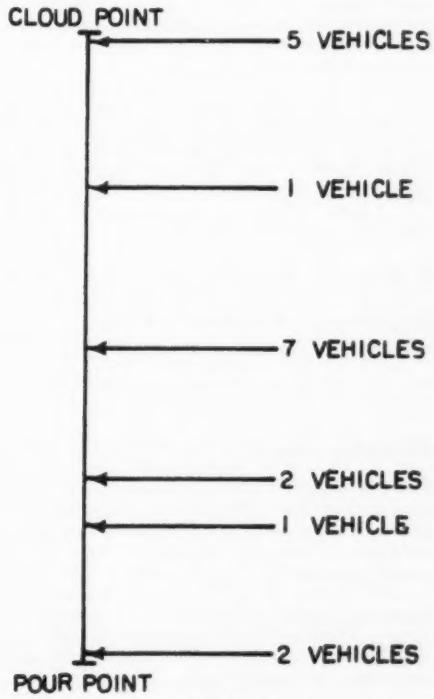

Fig K

LOW TEMPERATURE OPERABILITY LIMITS ON STRAIGHT-RUN DIESEL FUELS

the time wax crystals formed a serious build-up, sufficient heat was being supplied by the engine to melt the crystals. However, quite coarse filters on the suction side of the primary pump blocked, as did fine tubes and sharp elbows in tubes. It clearly appears, therefore, from these tests, that the vehicle manufacturer can do a lot to minimize effects of low temperature on fuel performance. In many cases an operability advantage of at least 10° F can be obtained by simple measures such as the removal or re-positioning of coarse strainers, the smoothing of sharp bends in pipes, and the use of wider bore tubing. The next point to emerge from the field

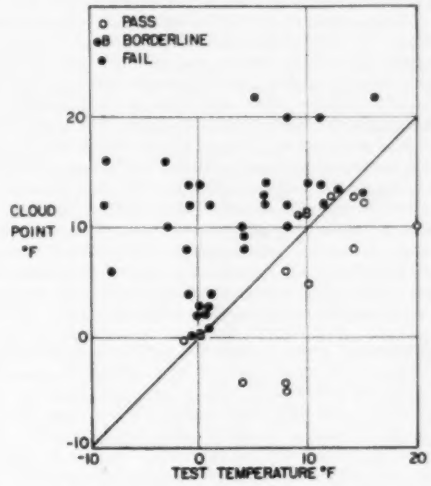

Fig L

LOW TEMPERATURE PERFORMANCE OF AUTODIESEL FUELS

Results of most critical vehicles

work was that once the failure temperature of a vehicle system had been established in terms of the cloud point and the pour point of a particular type of fuel, this failure temperature so expressed is independent of the fuel type, *i.e.* if a vehicle failed at the mean of the cloud and pour points, on a straight-run Middle East fuel, it would fail at about a temperature bearing the same relationship to cloud and pour points on a fuel containing cracked stocks, on a fuel from the Caribbean, on a fuel that had been dewaxed, or on a fuel that had been blended with kerosine. This point is illustrated in Fig L, where a number of results are given for fuels of all types in cloud point sensitive vehicles. It will be seen that the 1 : 1 cloud point correlation adequately separates the sheep from the goats, the passes from the failures. It therefore appears that from the point of view of providing satisfactory performance in practice, the cloud point test provides a simple and convenient test of operability of additive-free fuels.

The author presents a very helpful condensation of the published literature in the mechanism of pour depression, a field of much confusion and no little contradiction. And of course the cloud point is not a useful test for evaluating additives, since few do anything but raise it. In fact, it appears that nothing can replace field experiments. Certainly, in our experience, rig tests, although producing an interesting and satisfying wealth of data, are very disappointing when correlated with field experience.

Professor E. S. Sellers: I had the impression from the Shell Research papers that they were convinced that there was some breakdown of structure within their kerosine after shear had been applied. I think it was

Fig 2 of Mr Strawson's paper which shows that there is a considerable effect of that sort—that the waxy structure is broken down under shear and that what is left is the virtual Newtonian viscosity of the oil.

It was with considerable surprise that I heard later on that in the Esso results in pumping tests they found no difference at all, or only a few degrees of difference, between the temperature at which the suction side of the pump was blocked and that which blocked the discharge side. That seems to me to be remarkably inconsistent with what the first speakers had to say and I think that Mr Hutton felt that at one stage.

If the first speakers are right and the breakdown of structure does take place with the applied shear stress, then I wonder whether this is a real problem in aircraft. After all, I suppose that most of the tests were carried out with static tanks, but, in fact, once one gets the level of the tank down below absolutely full, how often is the oil in there absolutely static? Would there be sufficient agitation to give this an amount of shear sufficient to stop this structure building up, if the Shell Research people are right? On the other hand, if our friends from Abingdon are right, then presumably there is no breakdown of structure with increase of shear.

H. Strawson: I will answer the last part of the question first, because the influence of fuel agitation caused by slight rocking of the fuel tank is an aspect which we investigated in our full-scale tests. We found that the advantage to be gained from rocking by itself was so small as to be hardly worthwhile. I think that the amount of agitation received was insufficient to break down the structure appreciably. Furthermore, we were assured by the airline people that even very small amounts of rocking could not be relied upon at high altitudes. Incidentally, we did find that by leading some of the booster pump delivery back into a part of the tank remote from the pump, so that the fuel periodically received severe agitation within the booster pump itself, we obtained a considerable reduction in pumpability limit. This only worked satisfactorily in conjunction with a very slight degree of rocking—as low as $\pm \frac{1}{2}°$ at a frequency of 3 cycles/minute—to distribute the "treated" fuel throughout the tank. Although this would be a possible way of using fuels down to lower temperatures, it would of course involve some complication in the aircraft, not to mention a larger booster pump, which would not be readily acceptable by manufacturers and operators.

Answering Professor Sellers' first question, then, as to whether or not aviation kerosines are subject to breakdown of structure after shear has been applied, there is no doubt that they are. This might not be so marked with some kerosines as in the example whose rheological properties are shown in Fig 2, but the effect is always present.

I think that Professor Sellers was possibly mistaken in the apparent similarity between the results obtained upstream and downstream of the pump. If I have interpreted Mr Fowler's remarks correctly, flow downstream of the pump refers to the initiation of flow in a pipe within which the fuel has cooled statically. Nevertheless, I find it very surprising that Esso obtains a correlation between a dynamic laboratory test and flow upstream of the pump, where the fuel starts from a static condition and where one would expect the yield value to be the controlling factor.

Professor E. S. Sellers: Could we have a comment from Esso as to whether they really meant starting with the discharged pipe blocked?

L. B. Day: I should like to point out to Professor Sellers that we have obtained two correlations between laboratory tests and pumpability limits. The pumpability limit on the pressure side of the pump was found to correlate with the BP pumpability test and not with the break point in the viscosity curve, whereas the pumpability limit on the suction side of the pump correlated with this break point and not with the BP test.

S. C. Dodson: In our tests on the pressure side of the pump the fuel was cooled whilst stationary and the pump was switched on at intervals in order to see if the fuel had "solidified" or whether it was still pumpable.

J. W. Clarke: A number of previous speakers have stressed the importance of correlating rig tests with laboratory tests and I would like to know what is the intention of the working group of the IP General Test Panel when they have finished examining the methods put up by various oil companies. Is it the intention then to run another correlation programme with rig tests? I have a feeling that it would be very unpopular with a lot of people, particularly on the research side. However, on the other hand, it does seem a little odd to take methods, modify them, and they will undoubtedly be modified on the basis of my past experience of IP panels, and then to decide that the original correlations with rigs still apply.

Furthermore, Mr Fowler referred to plumbing intricacies of aircraft and it is probable that they will change in the future too.

I wonder if anyone on the working group is here and can speak on the earlier points. I would also like to ask if the IP are enlisting the co-operation of the ASTM in this matter. The ASTM already have specifications for jet fuels, based on cloud and pour point, and I know they are also working on pumpability test methods. It would be a great pity if we had one put up by the IP and a different one by the ASTM.

J. W. Hyde: The ASTM Committee has been formed to examine pumpability, and I know that BP has made the results of their studies available to ASTM. I would like to ask one very simple question on the rig tests. Has the aircraft vibration and its effect on fuel been taken into account, and would our rheological experts like to pass any observations on any possible effect?

H. Strawson: Yes, we have examined the effect of vibration of the aircraft tanks, hoping that this might give quite a large benefit. However, we were disappointed to find that although we applied vibrations at various frequencies, causing quite large standing waves on the surface of the fuel at normal temperatures, as the temperature approached the pumpability limit the waves merely died down; thickening and solidification proceeded as they would have done in the absence of any disturbance. Presumably the degree of agitation at any stage in the cooling process was insufficient to cause significant structure breakdown.

J. F. Hutton: Gavlin, Swire, and Jones [3] showed that the breakdown of structure, or decrease of viscosity, of a gelled oil is a regular function of the work done in shear; the greater the work done, the greater the decrease of viscosity. In these experiments the shearing was continuous. When the applied stress is cyclic, as in the case of a tank full of oil being vibrated in an aircraft, a similar relationship might be expected, provided the vibrations are dissipated by the gelled fuel. When the fuel has

been cooled so far that it has become virtually an elastic solid, the dissipation will be small and breakdown of structure will not occur, unless, of course, the amplitude and, perhaps, frequency of the vibration are increased. When the fuel gel is weak and more like a liquid, energy absorption can take place and the structure disrupt.

However, as Mr Strawson has said, the practical effect on fuel flow rate is small, so it is not wise to depend on aircraft vibrations to assist flow. Perhaps if the mode of vibration were suitably selected and variable some benefit might accrue, but the aircraft manufacturers would not look favourably on this approach.

J. W. Hyde: May I ask whether the vibrations were comparable in stress to those in practice?

H. Strawson: Yes, the maximum amplitude tried was chosen as being greater than would be experienced in practice.

W. H. Thomas: In reply to Mr Clarke's two queries, I would first observe that the working group, whose task will be to study methods for the assessment of flow properties, has only just been formed. What precise direction its deliberations will take we do not know at the moment, but it is of interest to know that the personnel of the working group will include two members from the aircraft industry.

The answer to the second query is that arrangements have already been made to keep ASTM fully informed of the deliberations and activities of the working group.

R. Piercy: May I first of all wish the working group every possible speed in reaching the point of actually putting an item in the specifications covering this matter, which is what the aircraft industry is mainly interested in.

I would like to mention one random point to start with —the question of vibration. Although it undoubtedly has some effect, we in the aircraft industry cannot of course expect it; we cannot guarantee that it will occur.

On the general question of specifications, one or two points arise. Would the pour point or pumpability point be an additional item to the F.P., or would it replace it? If it replaces it, could the F.P. go haywire? Would it be possible to have a pumpability of, say, −55 and a freezing point higher than the existing one of −50 or −40 according to the fuel? That might be of interest, because although at the moment fuel heating seems here to stay, it does entail a penalty, and any penalty is subject to constant investigation.

That brings me to the other random point: in the work at de Havilland on pumpability at low temperatures we have never encountered filter blockage by wax at temperatures above the visible cloud point and have therefore regarded the visual method of determining the cloud point as fairly reliable.

There are one or two other points here. If, when a pumpability item is included in the specification, a batch of fuel is found which does not comply, would the petroleum industry envisage putting in an additive, rejecting the batch, or balancing it with some other batch of fuel which has an abnormally low pumpability point? And if an additive is accepted as a remedy, would it be stable? Would there be any possibility of it weathering off, or anything like that?

With regard to Mr Dodson's contribution about the fuel freezing rigs at BP, the cooling of fuel in an integral tank in flight usually takes place from the bottom, from the tank floor, producing a temperature gradient through the depth of the fuel amounting to several degrees. Corresponding with this is a density gradient, which stops convection and causes stagnation within the fuel mass. Wax will form first along the tank floor, thickening upwards very slowly. From the photograph of the BP rig, it was evident that the test tank's vertical walls were subjected to the same cooling effects at the bottom. This would induce strong convection within the vessel and result in uniform and relatively fast cooling of the whole fuel. This could explain the uniform and thorough progress of solidification mentioned by Mr Dodson.

In actual flight conditions, for the reasons mentioned, we would expect a much slower cooling rate and more gradual growth of wax.

There was a related problem in the very interesting description of the DC.8 fuel system presented to the SAE recently; in tests done by Douglas the temperature fall of the fuel diminished sharply, very markedly, at the freeze point of the fuel, and they attributed this abrupt flattening of the cooling curve to the insulating properties of the wax forming on the tank floor.

We cannot find any evidence that the thermal conductivity of wax differs from that of the liquid fuel, and we believe that this flattening of the cooling curve is a function of the latent heat of solidification causing a temporary stabilization of temperature until the wax is fully formed, followed by a further drop. In that connexion, of course, we do feel that there is a big safety margin, in that it takes many hours to cool down anywhere near the pour point or the freezing point under cruising conditions, and undoubtedly it would take several more hours before a substantial quantity of the liquid could be converted into wax, owing to the very small temperature differential available to extract the latent heat of solidification from the liquid fuel.

I would like to know the authors' views on how soon the pour point is likely to go into the specification. We are most encouraged by the Esso results on viscosity break point and by the BP work in the same connexion.

H. Strawson: On the question of the additive, this was put forward only as a possibility at the present stage; so far as I am aware, nobody intends using an additive for this purpose in the near future, and clearly a great deal of research will be required on such effects as selectivity, compatibility, and stability. Therefore, as regards the action of a fuel company with a batch not meeting the pumpability specification, I can only say that for the near future, at any rate, it would not be able to supply it against that specification.

Regarding the distribution of temperature in the fuel as it cools, we have been very conscious of the complications this factor introduces into attempts at detailed theoretical analysis of the full-scale aircraft system and of the difficulties it makes in relating rig results to aircraft results. We have therefore tended to consider the worst case, *i.e.* when the whole of the tank contents are at the minimum temperature, and to treat non-uniformity of temperature as an alleviating factor which cannot generally be evaluated.

We have encountered the marked reduction of the rate of temperature fall when thickening of the fuel commences and attributed this not to a reduction in thermal conductivity but to the elimination of convention currents in the outer layers, both by the sides of the tank and by the vertical baffles which conducted heat away. I should have thought that in flight conditions the currents induced by aircraft movements, even if too small to prevent the formation of a wax structure, would be sufficient to distribute the temperatures so long as the fuel remains Newtonian.

In considering the effect of latent heat it must be remembered that only a small proportion of the fuel undergoes a change of phase, and also that we are not dealing with a pure compound but a mixture of a vast number of compounds, so that the wax formation does not occur at a fixed temperature.

S. C. Dodson: When we were making our tests we expected to find an insulating effect due to the "solid" layer and that its growth would be retarded. However, in our particular system, after the initial delay period, the thickness increased linearly with time up to the measured thickness of 3 inches. It appears that during the delay period there is some movement of the fuel which to some extent maintains the temperature of the bottom of the tank whilst the bulk temperature falls. Eventually, however, this movement is restricted and the fuel then solidifies on the tank walls and the rate of solidification is independent of the thickness already adhering. However, we are quite sure that our test does not represent all possible conditions in service.

Obviously the rate of heat flow from the bottom of the tank is a very important factor; if the temperature of the tank bottom is reduced quickly by more rapid heat extraction, the initial "solidification" would start earlier in the cycle and the subsequent rate of build-up would be affected also. The temperature might be reduced rapidly by blowing a high-speed draught over the tank but we were not in fact able to blow air at 600 m.p.h. in order to simulate the conditions in an integral wing tank. Work remains to be done to measure differences in cooling rates between the tests we have made, where the air was moving slowly, and the conditions in service, where air is scrubbing the surface of integral tanks at high speeds.

The Chairman then called for a vote of thanks to the authors, which was accorded with acclamation.

PERFORMANCE OF THE OLDERSHAW COLUMN AT REDUCED PRESSURE *

By S. R. M. ELLIS † and R. M. CONTRACTOR †

SUMMARY

A modified Oldershaw perforated plate column has been operated satisfactorily at pressures of 200 mm to 760 mm Hg abs for the foaming system methyl*cyclo*hexane–toluene and the non-foaming system ethylene-dichloride–toluene. The experimental results indicate that above the weepage point the overall efficiency of the column decreases at reduced pressures.

INTRODUCTION

THE Oldershaw [6] perforated plate column has in recent years become increasingly popular in laboratory distillations. Results obtained in 1-inch to 2-inch diameter batch or continuous Oldershaw columns are independent of preflooding, reproducible, and can be related to pilot and large-scale plate columns. In the literature little information exists on the performance of Oldershaw and plate columns at reduced pressures. Umholtz and van Winkle [7] report a decrease in the overall efficiency of a laboratory scale sieve tray column as the pressure is decreased. From tests on a plate column for pressures from 280 to 760 mm Marek [5] concluded that the influence of pressure on efficiency is negligible. Because information available on the influence of pressure on tray efficiencies is somewhat contradictory, it was decided to investigate the efficiency at different pressures of a six-tray modified Oldershaw column with hole diameter of 1·5 mm. This hole diameter was selected as previous work in this Department has shown that hole diameters of 0·9 and 1·1 mm were unsatisfactory at reduced pressures. Collins and Lantz [2] have also stated that an Oldershaw column with hole diameter of 0·9 mm is unsatisfactory at pressures below 250 mm Hg abs. In determining the efficiency at reduced pressures it was considered important that accurate vapour–liquid equilibrium results should be available, and therefore equilibrium data for the systems used were determined.

APPARATUS

A line diagram of the apparatus is shown in Fig 1. An ordinary 3-litre round-bottom flask (*A*), placed in an insulating jacket, was used as a still in conjunction with a 1-kW heater. Two ratemeters (*B*) and (*D*), similar to the one described by Ellis and Bahari [3] were used at the top and bottom of the column (*C*). Only the bottom ratemeter (*B*) was used for measuring the boil-up rate. The column was made of glass to facilitate visual observation of the flow mechanism. The column details are summarized in Table I. The column was surrounded with a length of 2-inch diameter glass tube, on the outer side of which a heating wire (280 Ω) was wound. This heating jacket was insulated by a 3-inch diameter glass tube. The

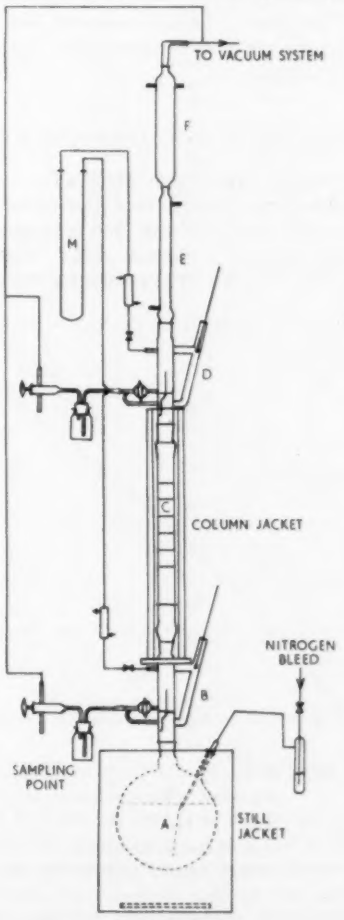

FIG 1
APPARATUS

* MS received 1 January 1959.

† Chemical Engineering Dept, The University, Birmingham.

heater wires were wound such that a required temperature gradient between the top and bottom of the column could be obtained. During the column operation, the temperature in the air jacket between the heater and the column was maintained about 1° C higher than the column temperature.

TABLE I

Details of the Modified Oldershaw Column

Diameter of column	30·5 mm
Plate spacing	30·0 mm
Diameter of perforations	1·5 mm
Number of perforations per plate	30
Percentage free area	7%
Number of actual plates	6

A 15-inch glass jacket condenser (*E*) mounted in series with an 11-inch coil and jacket condenser (*F*) were used. The pressure drop across the column was measured on a U-tube toluene manometer *M*. The desired pressure on the column was maintained by a controlled air bleed. Liquid sampling facilities were provided at the top and bottom of the column on the ratemeters (*B*) and (*D*).

VAPOUR–LIQUID EQUILIBRIUM RESULTS

Vapour–liquid equilibrium results for the system methyl*cyclo*hexane–toluene were determined experimentally at 760 mm, 500 mm, 350 mm, and 200 mm Hg abs pressures in a vacuum still.[4] These results are presented in Fig 2. The results for this near ideal

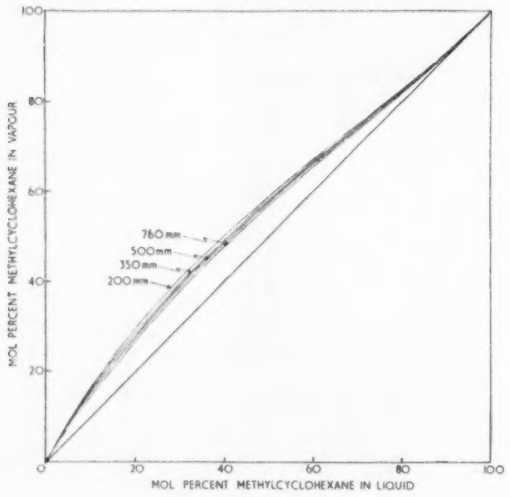

FIG 2

EQUILIBRIUM DATA FOR METHYL*CYCLO*HEXANE–TOLUENE SYSTEM

system correlated satisfactorily by the van Laar, Redlich-Kister, and Li-Coull equations. Equilibrium results of Colburn *et al.*[1] for ethylene–dichloride–toluene at 760 mm Hg abs pressure are given on Fig 3, together with results determined in the vacuum still [4] at 200 mm Hg abs pressure. For this ideal system the experimental results were in close agreement with those calculated by Raoult's Law.

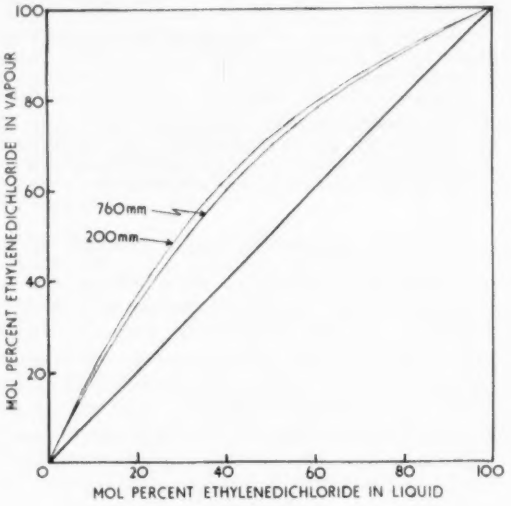

FIG 3

EQUILIBRIUM DATA FOR ETHYLENE–DICHLORIDE–TOLUENE SYSTEM

EXPERIMENTAL RESULTS AND DISCUSSION

All runs were carried out under conditions of total reflux. Overall efficiency results for methyl*cyclo*hexane–toluene and ethylene–dichloride–toluene at various boil-up rates and pressures are tabulated in Tables II and III. Samples of reflux at (*D*) (Fig 1) and the reflux leaving the column at (*B*) for both systems were analysed by measurement of refractive index of mixtures at 25° C using a Zeiss Abbe refractometer. The overall efficiencies were calculated by a McCabe–Thiele construction from the equilibrium results given in Figs 2 and 3. For methyl*cyclo*hexane–toluene the still composition was approximately 17·0 mol per cent methyl*cyclo*hexane, while for ethylene–dichloride–toluene the still composition was approximately 7·0 mol per cent ethylene dichloride.

The effect of pressure and boil-up rate on overall efficiency are shown on Figs 4 and 5, where the overall efficiency is plotted against the *F* factor, where the *F* factor is the product of linear vapour velocity (*U*) and the square root of vapour density (ρ_v). It is considered that efficiency and pressure drop results at different pressures are best correlated by the use of the *F* factor. The absolute pressure at the top of the column is the parameter. From these figures it will be seen that for both systems, when the column is operating above the weepage point, the overall efficiency decreases with a decrease in column pressure

TABLE II

Overall Efficiency Results—Methylcyclohexane–Toluene

Pressure on the column	Boil-up rate G lb/hr, sq ft	Vapour velocity, U ft/sec.	F factor $U\sqrt{\rho_v}$	Overall efficiency %
Atm	100·5	0·15	0·064	91·0
,,	125·7	0·19	0·080	90·0
,,	185·1	0·27	0·118	87·0
,,	186·1	0·27	0·119	85·1
,,	236·5	0·35	0·151	84·6
,,	275·0	0·41	0·176	81·5
,,	340·0	0·50	0·217	76·1
,,	342·0	0·51	0·219	76·35
,,	367·0	0·54	0·234	76·5
,,	372·0	0·55	0·238	76·5
,,	385·2	0·57	0·247	77·0
,,	419·5	0·62	0·268	81·0
,,	486·0	0·72	0·310	82·0
,,	556·0	0·82	0·356	79·5
,,	608·0	0·90	0·388	78·3
,,	620·5	0·92	0·398	76·7
,,	691·0	1·02	0·441	75·6
,,	700·0	1·03	0·447	73·6
,,	813·5	1·20	0·518	69·0
,,	863·0	1·42	0·615	65·5
500 mm Hg abs	74·5	0·16	0·058	92·0
,, ,,	158·5	0·34	0·122	86·0
,, ,,	161·9	0·35	0·125	88·0
,, ,,	189·0	0·41	0·146	87·0
,, ,,	238·7	0·51	0·184	80·6
,, ,,	325·8	0·70	0·251	74·2
,, ,,	378·0	0·81	0·292	77·4
,, ,,	469·0	1·01	0·362	75·2
,, ,,	586·0	1·20	0·431	71·8
,, ,,	642·5	1·39	0·496	69·7
,, ,,	781·0	1·69	0·603	66·2
350 mm Hg abs	115·0	0·34	0·103	89·3
,, ,,	181·0	0·54	0·162	86·0
,, ,,	203·6	0·61	0·186	79·0
,, ,,	208·0	0·62	0·187	77·5
350 mm Hg abs	224·0	0·66	0·201	76·0
,, ,,	248·0	0·74	0·223	75·0
,, ,,	384·0	1·14	0·344	72·0
,, ,,	384·0	1·14	0·344	72·4
,, ,,	455·0	1·35	0·408	72·0
,, ,,	540·0	1·60	0·485	66·5
,, ,,	617·5	1·83	0·555	63·2
200 mm Hg abs	89·5	0·44	0·105	88·0
,, ,,	121·2	0·60	0·141	95·8
,, ,,	141·5	0·70	0·166	89·2
,, ,,	148·3	0·74	0·175	85·0
,, ,,	167·9	0·83	0·197	80·1
,, ,,	180·3	0·90	0·212	76·9
,, ,,	183·0	0·91	0·215	75·0
,, ,,	193·6	0·96	0·226	75·1
,, ,,	241·8	1·20	0·284	71·5
,, ,,	313·2	1·56	0·368	70·2
,, ,,	331·2	1·68	0·396	67·2
,, ,,	397·2	2·01	0·475	66·2
,, ,,	438·5	2·18	0·516	65·2
,, ,,	465·5	2·32	0·547	64·6
,, ,,	492·2	2·45	0·578	63·5
,, ,,	492·2	2·45	0·578	63·2
,, ,,	499·2	2·53	0·597	63·5
,, ,,	544·0	2·71	0·638	63·5

and with an increase in boil-up rate. The F factors corresponding to the weepage and flood points are almost unaffected with a change in column pressure.

Below the weepage point the overall efficiency increases with a decrease in boil-up rate. When the plates start weeping, the liquid, after passing through the holes, travels underneath the plate and flows down the column wall to the plate below. It has been observed that the weeping is intermittent and the resulting pulsation effect causes ripples on the liquid film on the walls. This pulsating wet wall effect appears to be more effective at decreasing boil-up rates.

TABLE III

Overall Efficiency—Ethylene–Dichloride–Toluene System

Pressure on the column	Boil-up rate G lb/hr, sq ft	Vapour velocity, U, ft/sec.	F factor, $U\sqrt{\rho_v}$	Overall efficiency %
Atm	156·0	0·23	0·100	75·0
,,	232·0	0·34	0·149	71·0
,,	276·0	0·41	0·177	67·3
,,	331·0	0·49	0·212	63·0
,,	340·0	0·50	0·218	63·1
,,	423·0	0·63	0·272	62·5
,,	575·0	0·85	0·368	61·1
,,	660·0	0·98	0·424	60·4
,,	805·0	1·19	0·515	58·2
,,	940·0	1·39	0·602	57·5
,,	1240·0	1·84	0·795	57·4
200 mm Hg abs	166·0	0·84	0·196	68·0
,, ,,	226·0	1·14	0·266	61·5
,, ,,	276·0	1·40	0·327	59·9
,, ,,	317·0	1·51	0·353	58·6
,, ,,	384·0	1·95	0·455	58·1
,, ,,	398·0	2·02	0·472	58·5
,, ,,	435·0	2·20	0·514	56·5
,, ,,	527·0	2·68	0·626	55·15
,, ,,	607·0	3·08	0·720	55·0

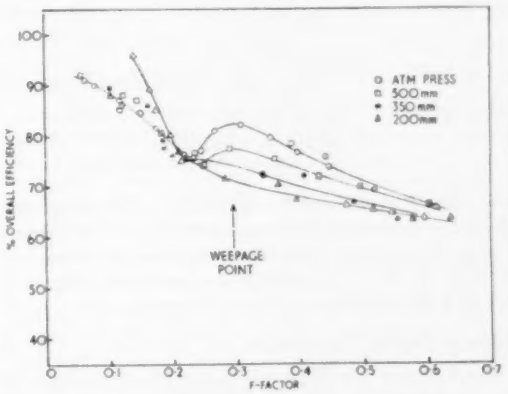

FIG 4

OVERALL EFFICIENCIES:
SYSTEM METHYL*CYCLO*HEXANE–TOLUENE

From Fig 5 it can be seen that above the weepage point for ethylene–dichloride–toluene the decrease in efficiency with pressure is independent of the boil-up rate. For methyl*cyclo*hexane–toluene the decrease in efficiency varies with the boil-up rate. This difference in column performance is thought to be due to the difference in foaming characteristics of the two systems.

Zuiderweg and Harmens [8] have reported that a stable foam is produced on a plate column when the linear vapour velocity is low, when the difference in concentration between the operating line and equilibrium line is large, and when the surface tension of the reflux increases down the column. Any such system where the surface tension increases down the column is referred to as a positive system, while when the surface tension of the reflux decreases the system is a negative or non-foaming system. Methyl*cyclo*hexane–toluene is a positive system, while ethylene–dichloride–toluene is a negative.

In the case of methyl*cyclo*hexane–toluene at 760 mm, the rapid decrease in efficiency with an increase in the F factor is because of the decrease in contact time and the change in foam structure with increasing vapour velocity. At 200 mm Hg abs pressure, even at a low F factor, the linear vapour velocity is too high to give a stable foam and thus an increase in F factor

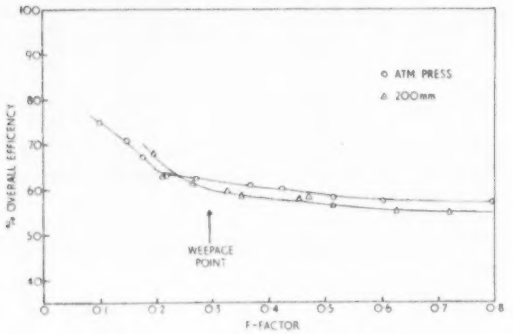

FIG 5

OVERALL EFFICIENCIES: SYSTEM ETHYLENE–DICHLORIDE–TOLUENE

only slightly decreases the efficiency. For the system ethylene–dichloride–toluene, where the surface tension change is negative and little or no foam is formed, an increase in F factor slightly decreases the efficiency, due to a decrease in the time of contact.

Effect of Liquid Composition

Fig 6 shows the influence of liquid composition on the overall efficiency. For the system methyl*cyclo*hexane–toluene, at atmospheric pressure it is noticed that at extreme concentrations the column efficiency decreases owing to the decreased amount of foam formed, due to the low concentration difference between the equilibrium curve and operating line. At 200 mm Hg abs pressure there is no marked influence of composition on efficiency, as the higher linear vapour velocity at reduced pressure gives little or no foam formation on the plates.

Individual Film Resistances

The effect of pressure on individual film resistances for the system ethylene–dichloride–toluene is shown on Fig 7, where the reciprocal of the number of overall vapour transfer units $\left(\frac{1}{N_{OG}}\right)$ is plotted against the average slope of the equilibria curve "m_a" for a constant F factor of 0·6. The number of overall

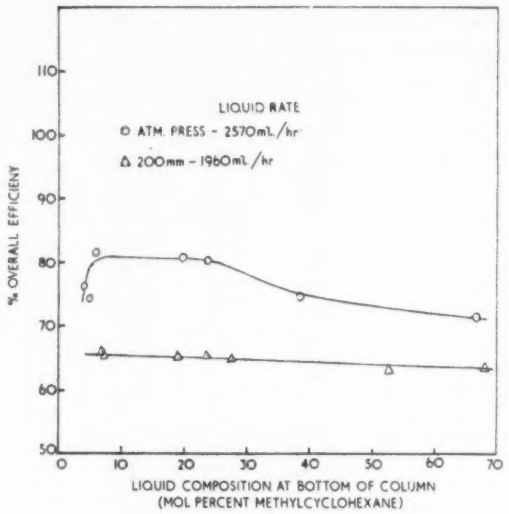

FIG 6

EFFECT OF CONCENTRATION ON EFFICIENCY: SYSTEM METHYL*CYCLO*HEXANE–TOLUENE

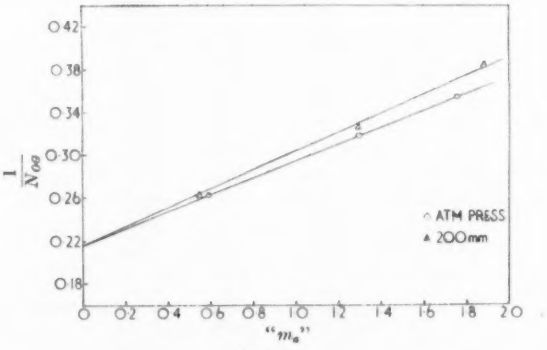

FIG 7

RECIPROCAL OF OVERALL VAPOUR TRANSFER UNITS *v.* SLOPE OF EQUILIBRIUM CURVE: AT F FACTOR = 0·6, SYSTEM ETHYLENE–DICHLORIDE–TOLUENE

transfer units based on vapour phase were calculated by graphical integration of the equation

$$N_{OG} = \int_{y_{n+1}}^{y_n} \frac{dy}{y^* - y} \quad . \quad . \quad . \quad (1)$$

From the straight line graphs it is noted that the vapour film resistance is unaffected by pressure and that as the slope of the line for 200 mm pressure is a little higher than at 760 mm, the liquid film resistance has increased by 13·0 per cent. At 760 mm the

system ethylene–dichloride–toluene has a vapour film resistance equal to 73·0 per cent of the total resistance to mass transfer at "m_a" equal to unity.

The reason for applying the equation

$$\frac{1}{N_{OG}} = \frac{1}{N_G} + \frac{m\,G/L}{N_L} \quad . \quad . \quad . \quad (2)$$

was the assumption that for the non-foaming negative system ethylene–dichloride–toluene the interfacial

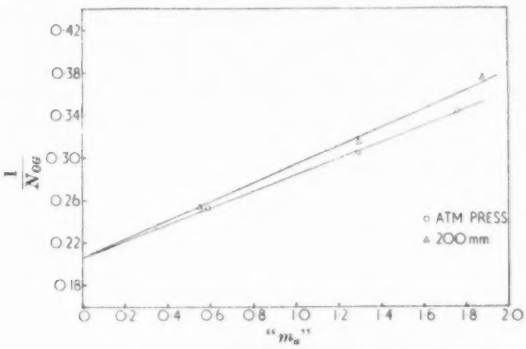

Fig 8

RECIPROCAL OF OVERALL VAPOUR TRANSFER UNITS *v.* SLOPE OF EQUILIBRIUM CURVE: AT *F* FACTOR = 0·4, SYSTEM ETHYLENE–DICHLORIDE–TOLUENE

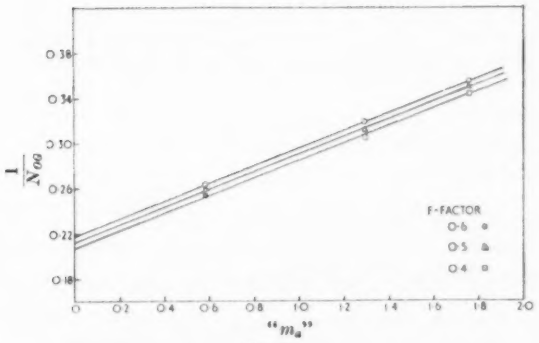

Fig 9

RECIPROCAL OF OVERALL VAPOUR TRANSFER UNITS *v.* SLOPE OF EQUILIBRIUM CURVE: AT ATMOSPHERIC PRESSURE, SYSTEM ETHYLENE–DICHLORIDE–TOLUENE

area of contact was relatively constant at a constant *F* factor.

Fig 8 shows a further plot of $\left(\frac{1}{N_{OG}}\right)$ against "m_a" at an *F* factor of 0·4 and again it is established that the vapour film resistance is unaffected by pressure, while the liquid film resistance increases by 13·0 per cent as the pressure is reduced from 760 to 200 mm. As the system is mainly vapour, film controlling the increase in liquid film resistance has little effect on the overall efficiency.

Fig 9 indicates that for ethylene–dichloride–toluene

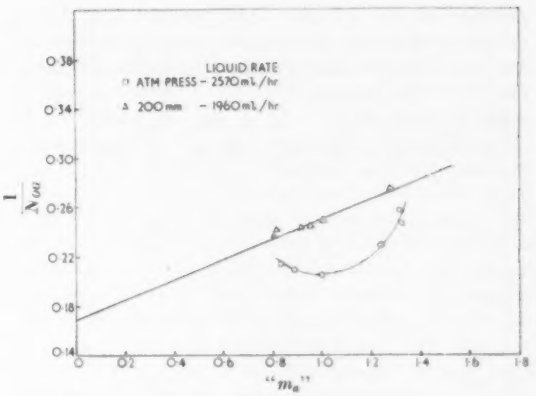

Fig 10

RECIPROCAL OF OVERALL VAPOUR TRANSFER UNITS *v.* SLOPE OF EQUILIBRIUM CURVE: SYSTEM METHYL*CYCLO*HEXANE–TOLUENE

at atmospheric pressure, although the vapour film resistance decreases with an increase in the *F* factor, the liquid film resistance is independent of the boil-up rate.

For the positive foaming system methyl*cyclo*hexane–toluene the results on Fig 10 for atmospheric pressure show that equation (2) does not apply. At 200 mm, because of the absence of foam on the plates, a straight line plot is obtained giving for methyl*cyclo*hexane–toluene a vapour film resistance of 68·0 per cent at "m_a" equal to unity.

Pressure Drop Results

The total pressure drop through the column increases with an increase in boil-up rate and with a

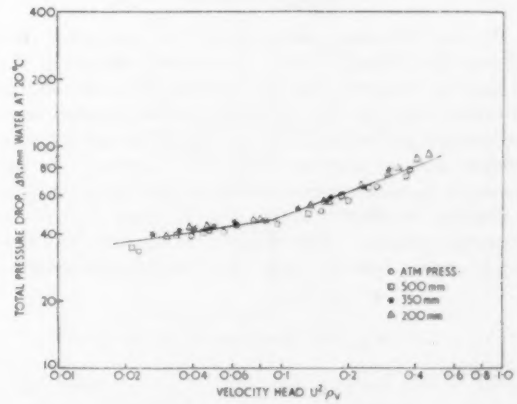

Fig 11

PRESSURE DROP RESULTS: SYSTEM METHYL*CYCLO*HEXANE–TOLUENE

decrease in absolute pressure on the column. It is found that the total pressure drop at a constant *F* factor is for a given system nearly independent of the

pressure on the column. The pressure drop for the ethylene–dichloride–toluene is a little higher than that for methyl*cyclo*hexane–toluene at a constant F factor. On Fig 11 the pressure drop ΔP is plotted against the velocity head $U^2 . \rho_v$ on a log–log paper for the system methyl*cyclo*hexane–toluene. The graph gives two straight lines intersecting at a value of $U^2 . \rho_v$ of

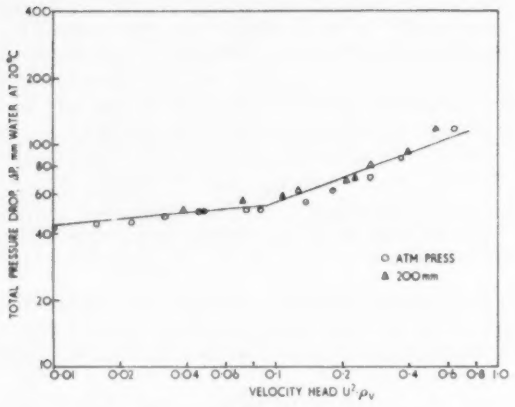

FIG 12

PRESSURE DROP RESULTS:
SYSTEM ETHYLENE–DICHLORIDE–TOLUENE

approximately 0·09, corresponding to an F factor of 0·3, the point at which the plates start weeping. A similar plot is shown on Fig 12 for ethylene–dichloride–toluene, with the break again at a point corresponding to the weepage point.

CONCLUSIONS

For an Oldershaw plate column it is concluded that above the weepage point the overall efficiency decreases at reduced pressures and that the order of this decrease is greater for a foaming positive system such as methyl*cyclo*hexane–toluene than for a non-foaming system such as ethylene–dichloride–toluene. As the pressure is reduced the resulting high linear vapour velocities decrease the amount of foam formed in foaming systems. Also the greater decrease in efficiency with boil-up rate for methyl*cyclo*hexane–toluene compared with ethylene–dichloride–toluene is due to the change in foam structure in the former system with increasing vapour velocity.

For non-foaming mixtures in an Oldershaw column it is possible for a given boil-up rate to evaluate gas and liquid film efficiencies by plotting the reciprocal of the number of overall transfer units against the average slope of the equilibrium curve. At 760 mm the vapour film resistance is 73·0 per cent of the total resistance for the system ethylene–dichloride–toluene, while at 200 mm for methyl*cyclo*hexane–toluene at approximately the same F factor the vapour film is 68·0 per cent of the total resistance. For ethylene–dichloride–toluene the vapour film resistance appears to be independent of pressure, while the liquid film resistance increases by 13·0 per cent as the pressure is reduced from 760 to 200 mm.

NOTATION

F	F factor based on column cross-sectional area, $F = U . \sqrt{\rho_v}$.
G	Vapour mass rate, lb/(hr) (sq ft).
L	Liquid mass rate, lb/(hr) (sq ft).
m	Slope of equilibrium curve.
"m_a"	Average slope of equilibrium curve for a range of composition.
N_G	Number of gas phase transfer units.
N_L	Number of liquid phase transfer units.
N_{OG}	Number of overall transfer units based on gas phase.
ΔP	Total pressure drop across the column, mm of water at 20° C.
U	Superficial linear vapour velocity ft/sec.
x	Mol per cent of light component in liquid phase.
y	Mol per cent of light component in vapour phase.
y^*	Mol per cent of vapour in equilibrium with x.
ρ_v	Vapour density, lb/cu ft.

Subscripts

n, $n + 1$ Plate numbers.

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THE ASTM COLOUR SCALE

In the 1955 Edition of ASTM Standards there appeared a Proposed Method of Test for Measuring the Colour of Petroleum Products (ASTM Colour Scale). This was, in fact, an improved version of the ASTM–Union Method (D155) that has been in use for over thirty years. The status of the new method was raised to Tentative in the 1957 Edition, where it appeared under the designation D1500–57T with the recommendation that it should replace Method D155 in 1958. Withdrawal of Method D155 has since been postponed.

Although the ASTM–Union method has not been adopted by the Institute of Petroleum, it is widely used in the U.K. and several members of the Institute have expressed interest in the current changes. The following notes have, therefore, been produced for their information.

The method for measuring colour of petroleum products by means of the ASTM–Union Colorimeter was first adopted by the ASTM in 1923 and has continued with only relatively minor changes until the present day. It has, however, never been regarded as entirely satisfactory and has therefore remained only a "Tentative" method. In recent years, Research Division IX of ASTM Committee D2 have given a great deal of attention to the problem of overcoming three of the basic shortcomings of the method. These are:

1. The standard glasses are not defined in absolute units; as a result there are wide variations between the glasses in use in different laboratories.
2. The chromaticities (*i.e.* the "hues" as expressed in terms of "redness," "yellowness," etc.) of the lighter glasses do not correspond very well with present-day oils.
3. The scale is uneven, *i.e.* the difference in chromaticity (spacing) between successive glasses is not uniform.

Details of this work have been given in the *Journal of Research of the National Bureau of Standards*,* and the new scale that was developed was described in the *ASTM Bulletin*.† This new scale is similar to the old in that it covers the same range with standard glasses numbered from 1 to 8, but there are important differences:

1. The new glasses are defined in terms of their chromaticity and their luminous transmittance.
2. The characteristics of the lighter glasses have been modified so that they are a better match for present-day oils.
3. The scale has been replaced so that the steps between successive glasses are more uniform.

In the course of revising the method, the opportunity was taken to improve the colorimeter itself with the result that it is not possible to convert an existing instrument simply by fitting new glasses. The approximate relationship between readings on the two scales is shown in Fig 1, and this may be used as a conversion chart provided that a high degree of accuracy is not required. For greater precision it is preferable to use the appropriate instrument, and for arbitration purposes it is necessary to use glasses that have been certified as conforming to the specifications.

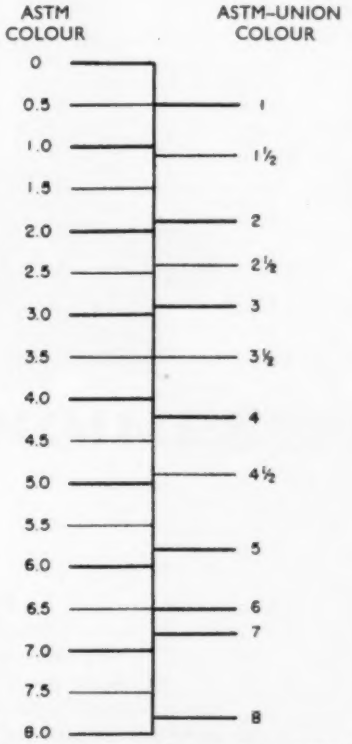

Fig 1: See ASTM Standards 1955, P 94–D, Fig 2.

Measurements by the new method should be reported as "ASTM" colours to distinguish them from the ASTM–Union colours. During the period that the two methods are in use, it is recommended that the distinction be emphasized by adding "D1500" (or D155 for the ASTM–Union Method).

* Judd, D. B., *et al. J. Res. nat. Bur. Stand.*, 1950, **44**, RP2103.

† Hancock, H. M., and Watt, J. J. *Bull. Amer. Soc. Test. Mat.*, 1954, (201).

OBITUARY

GEORGE WILMOT INGLE NOKES

1904–1959

THE death on 16 February 1959 of G. W. I. Nokes is announced with regret. An Associate Member of the Institute since 1937, his first entry into the petroleum industry was when he joined the Medway Oil and Storage Co. Ltd as a student apprentice at their plant on the Isle of Grain. Two years later he was appointed the Company's shipping and storage manager.

In 1932 he joined Berry Wiggins and Co. Ltd as assistant refinery superintendent at Kingsnorth, Kent. Later he moved to the head office in London as assistant to the general manager on engineering and refinery matters.

He remained with Berry Wiggins until 1945, when he became senior sales engineer to Peabody Ltd. Later he was made general manager of the Company, the position he held at the time of his death.

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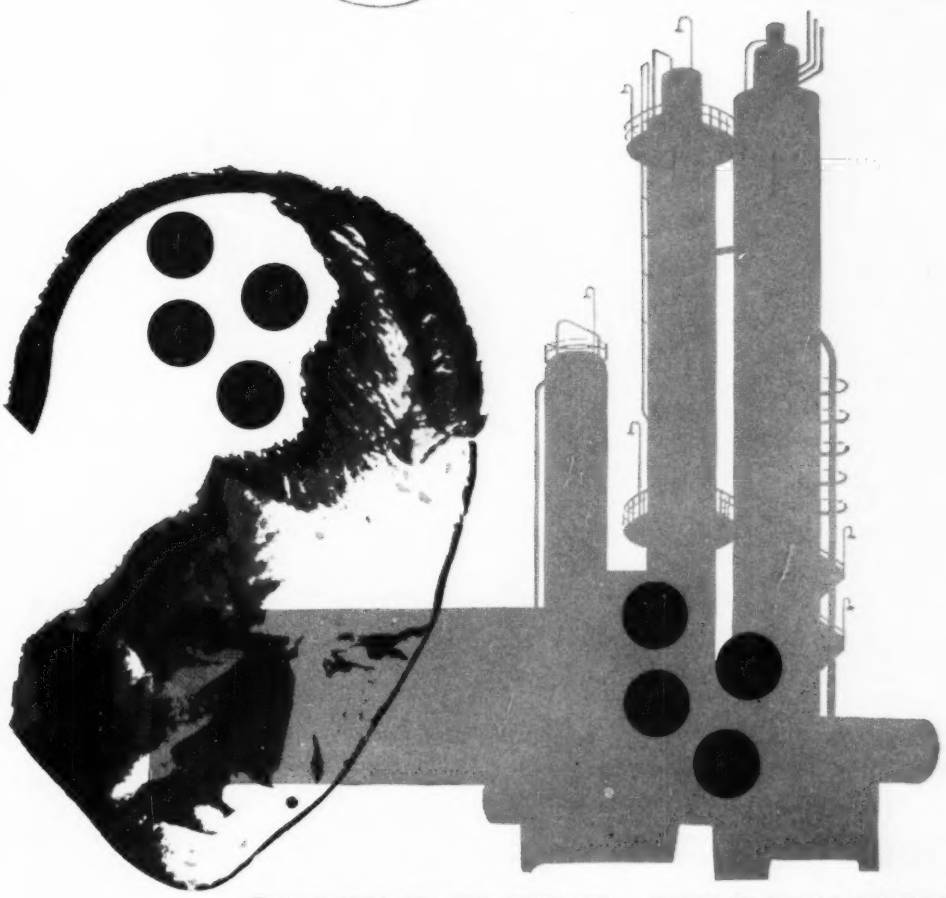

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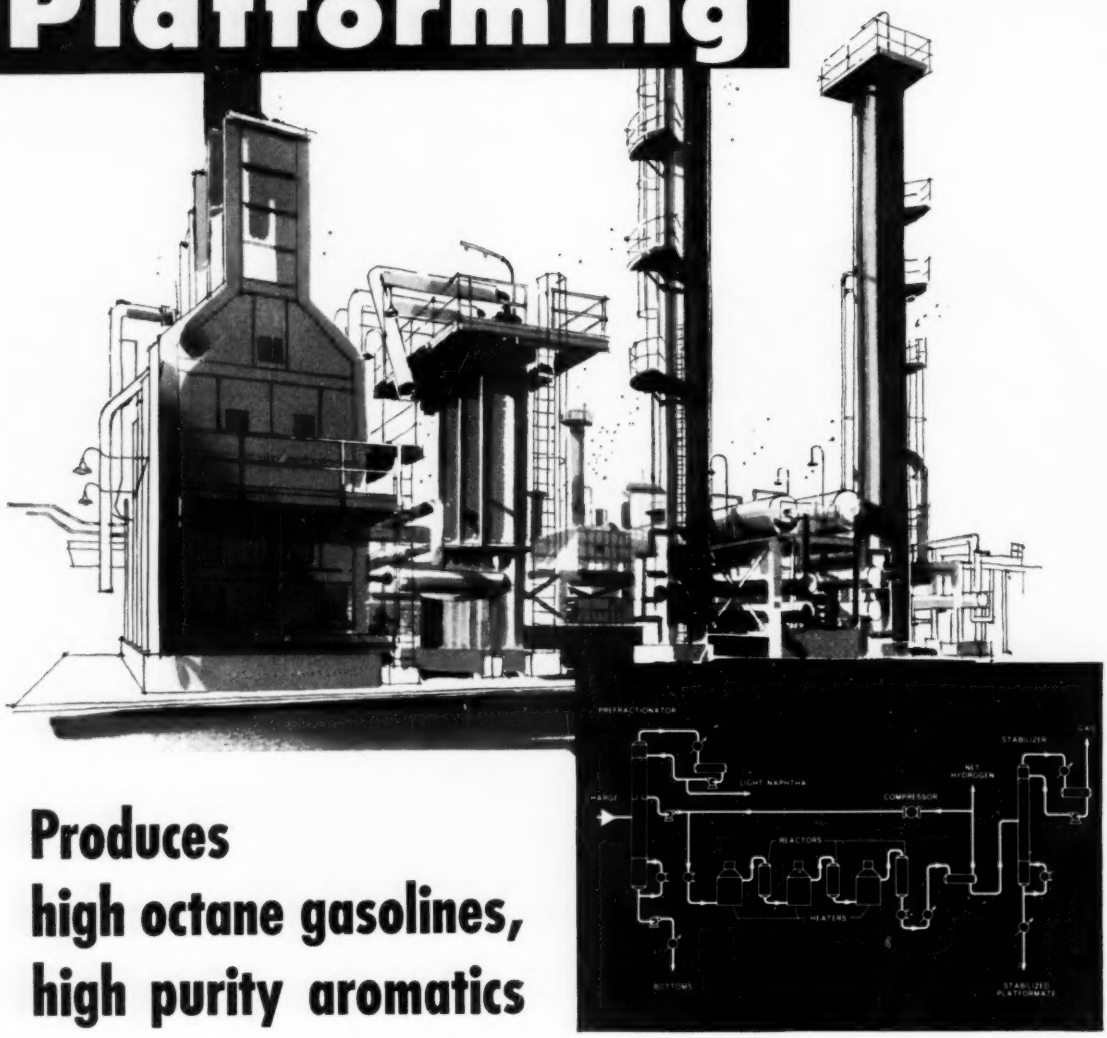

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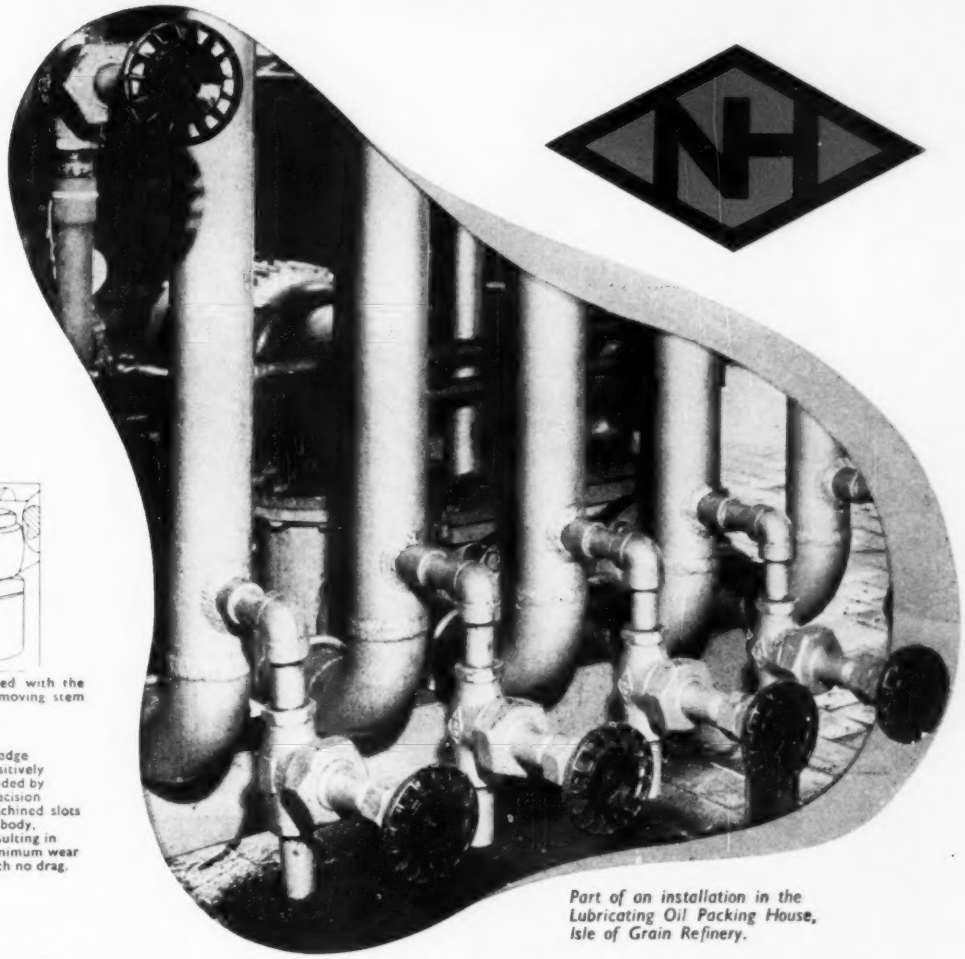
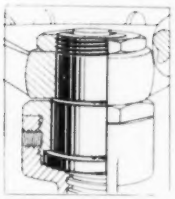


Part of an installation in the Lubricating Oil Packing House, Isle of Grain Refinery.



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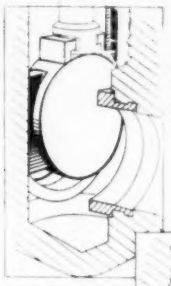

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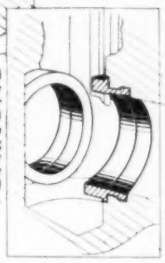

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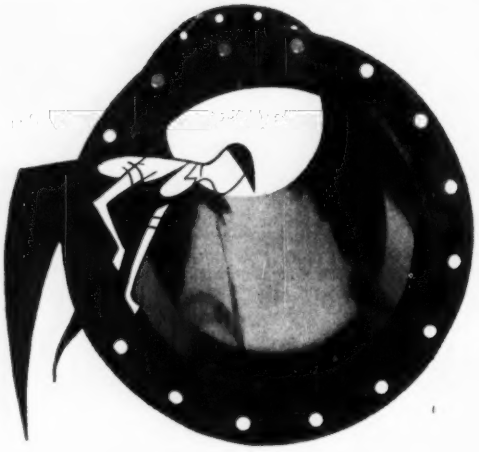

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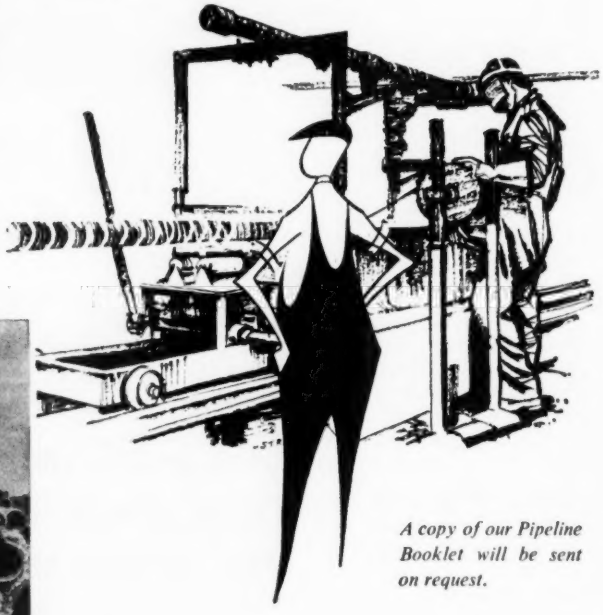

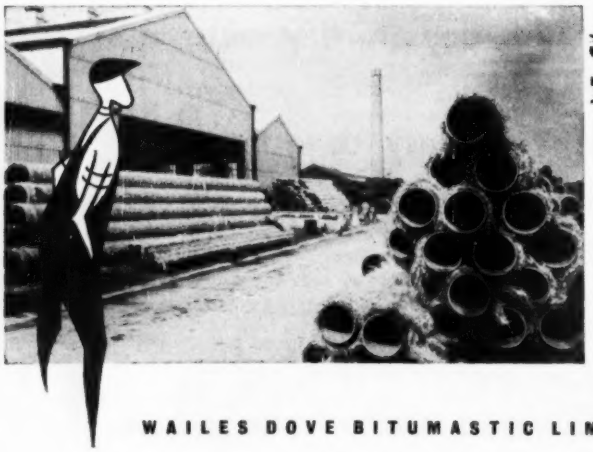

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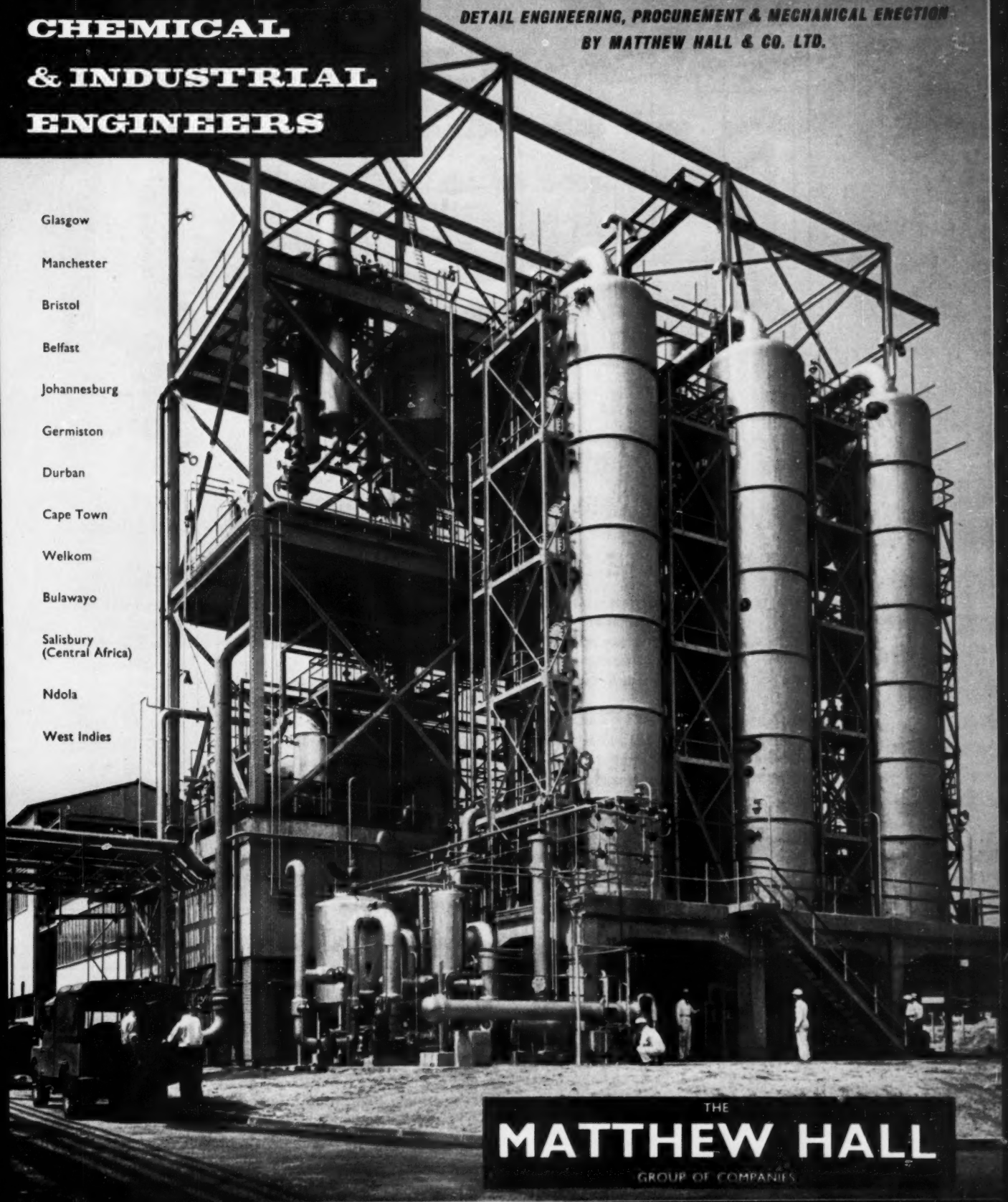

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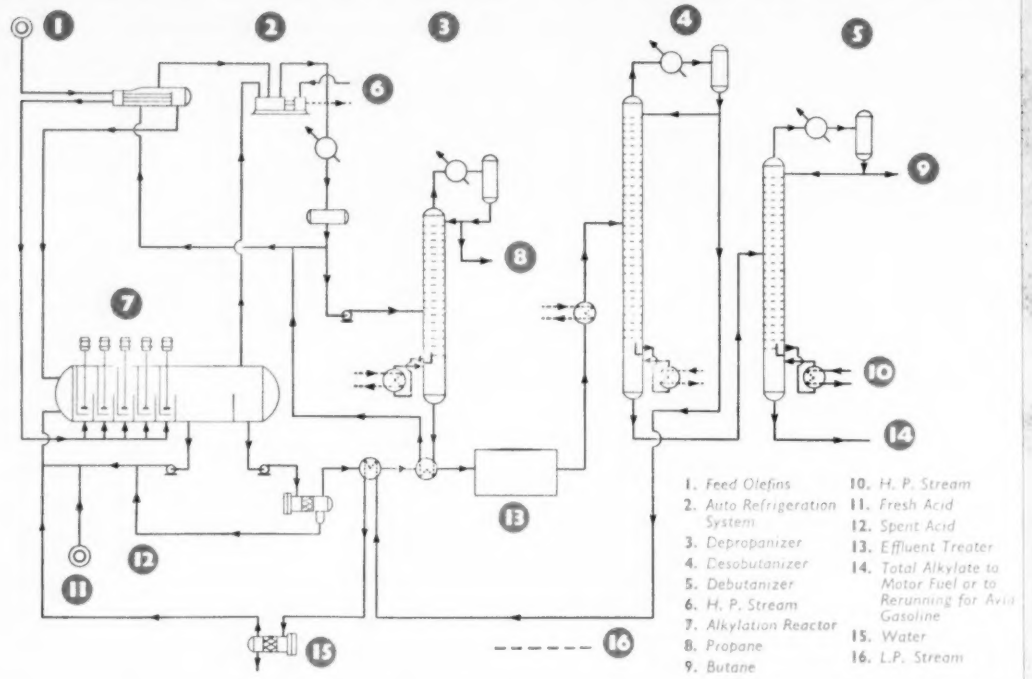

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The Kellogg cascade reactor as shown is a five zone reactor with an internal emulsion pump for each zone and auto-refrigeration. Five streams of olefin feed flow into the five separate zones while the isobutane recycle and acid flow in series through these zones. This arrangement creates a high isobutane concentration and a high isobutane-olefin ratio in each stage, thereby establishing optimum conditions for maximum production of high quality alkylate with a minimum quantity of isobutane recycle.

Acid consumption has been reduced by recent improvements in the reactor design and by utilizing spent acid recirculation plus the use of oleum as part of the fresh acid make-up. Another advantage is as the octane number is increased the acid consumption is decreased. Most of the corrosion problems associated with sulphuric acid alkylation have been eliminated as a result of recent design innovations. Refiners will find this route blazed with the experience of designing over thirty alkylation units, the best way to achieve better quality petrol. Kellogg International Corporation welcomes the opportunity to discuss alkylation with interested refinery engineers.

TYPICAL ALKYLATION YIELD DATA

Olefin Feed Type—	Propylene	40% C_3 60% C_4	Butylenes	Pentenes
Yield—Bbl. Alkylate per Bbl. Olefin	1.78	1.74	1.72	1.60
Isobutane Consumption—Bbl. iC_4 per Bbl. Olefin	1.275	1.174	1.106	0·965
Avg. Acid Consumption: Lbs. 98% H_2SO_4 per Gal. Tot. Alkylate	2.5.0.84	1.5.0.6	0.84.0.33	1.0.0.4
338°F End Point Alkylate Quality				
F.1 Research Octane No., Clear	89.92	92.95	94.97	90.93
F.1 Research Octane No., +3.0cc TEL (WEISE SCALE)	101.9-104	104-107	106.8-109.3	102.6-104.9
F.2 ASTM Octane No., Clear	87.90	90.93	92.94	90.92
F.3 or I.C. Performance No., 4.6cc TEL	117.122	124.127	127.132	116.123
F.4 or 3.C. Performance No., 4.6cc TEL	129.142	141.6.154	150.162	136.148

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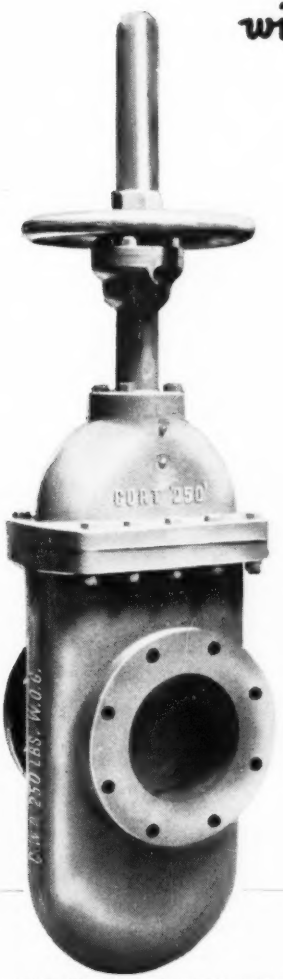

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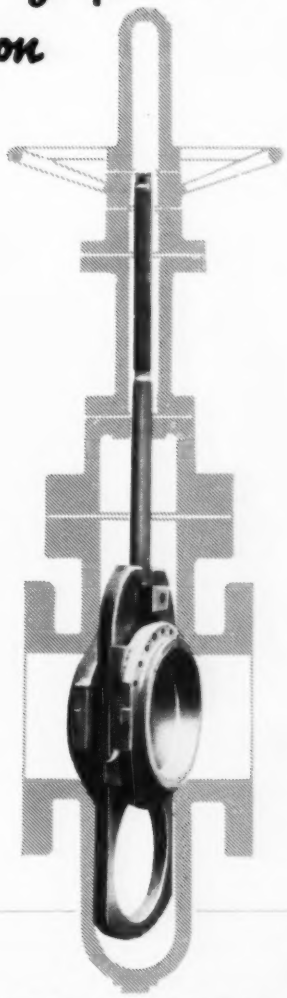

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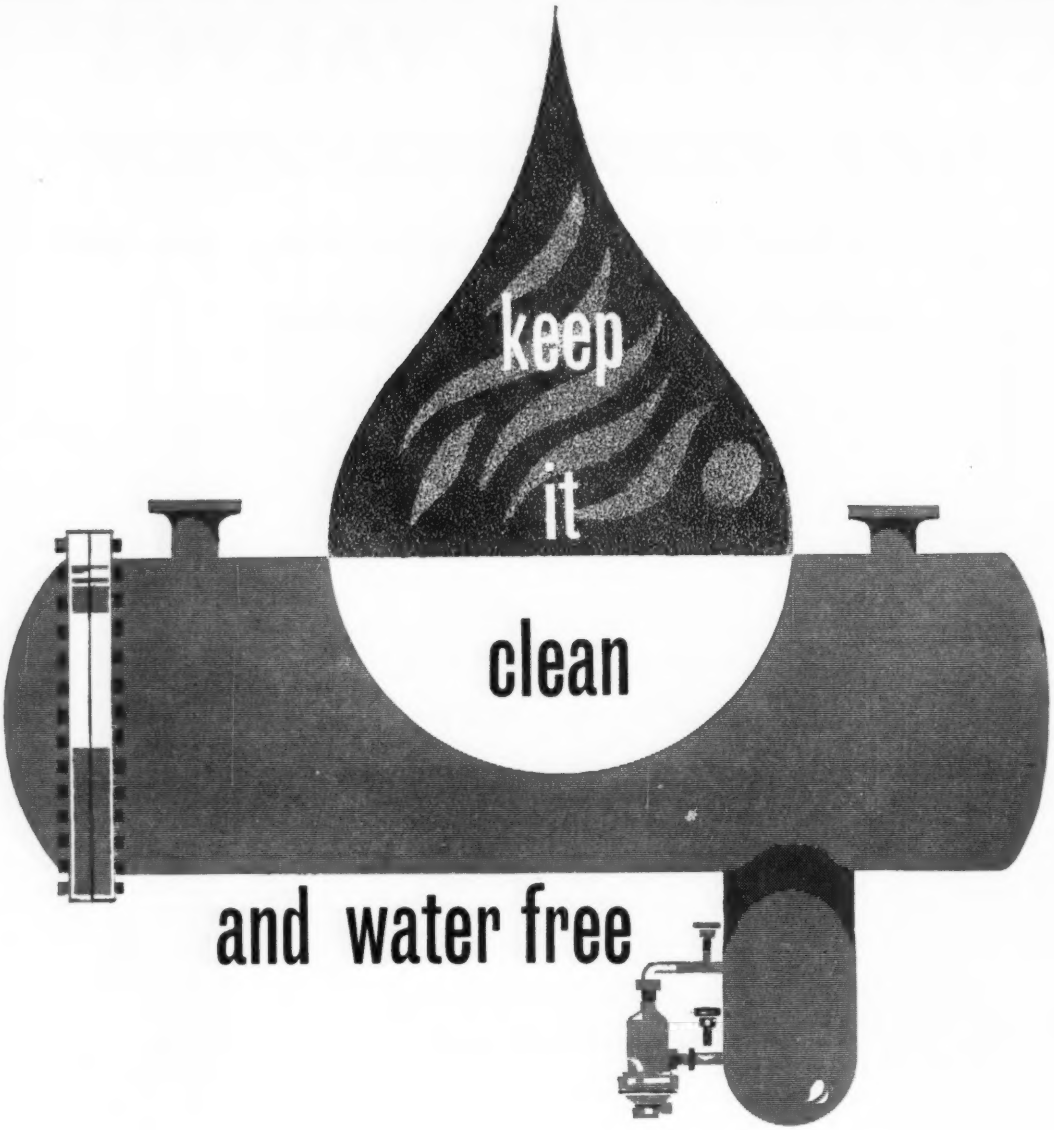

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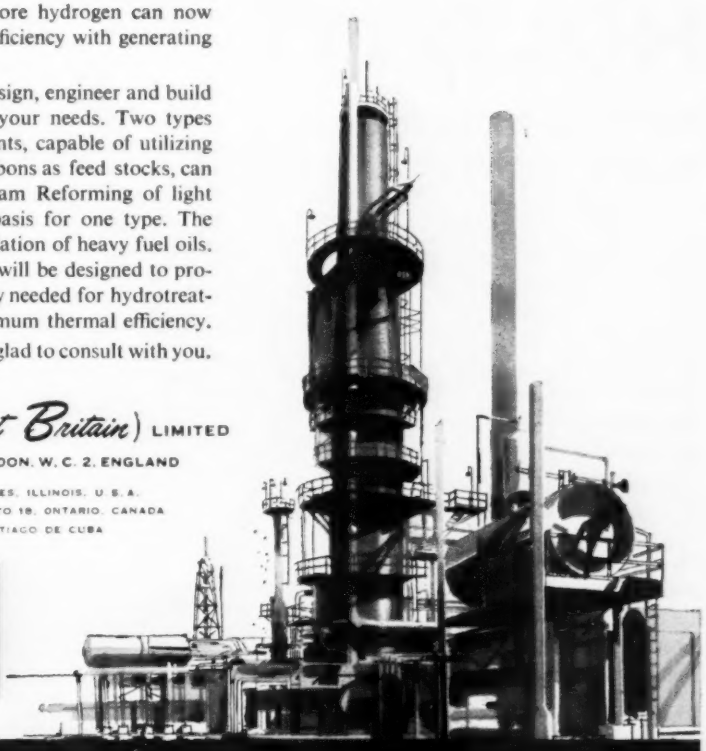

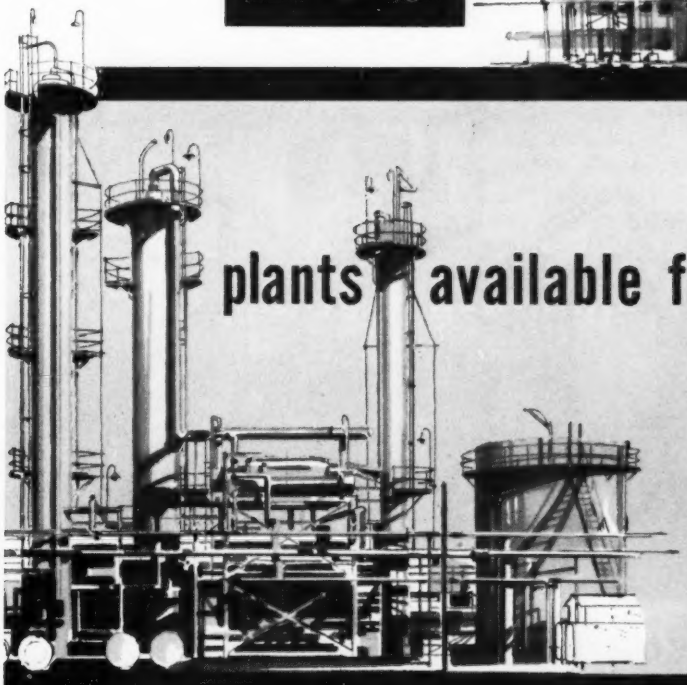

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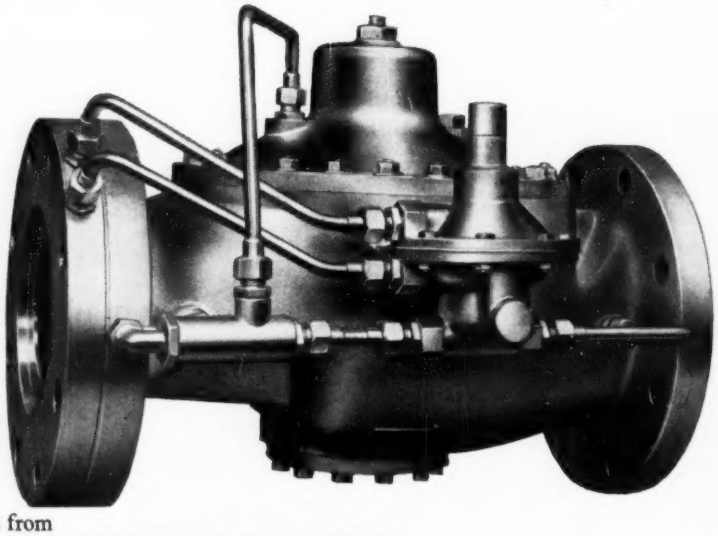

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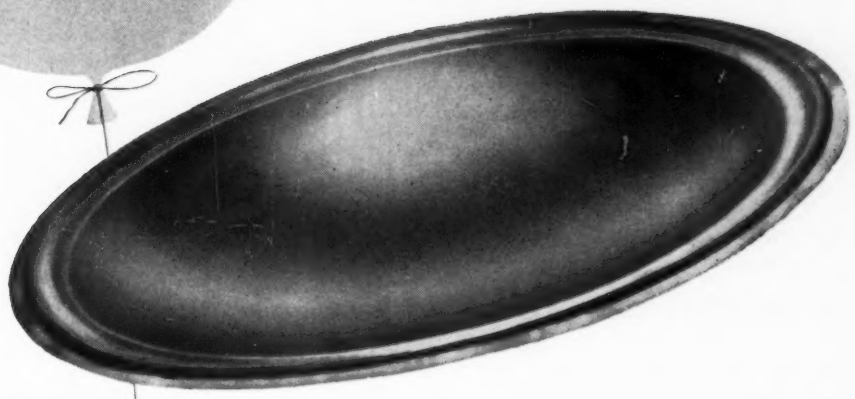

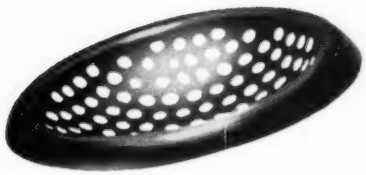

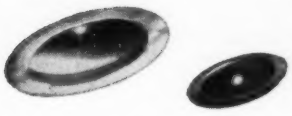

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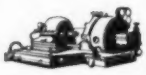

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